Revised (2)

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Supporting Information

Efficient Stereochemical Relay En Route to Leucascandrolide A

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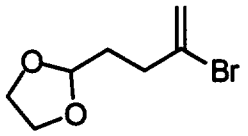
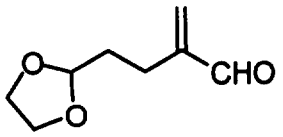
ether (200 mL) and water (200 ml). The organic layer was separated, washed with water (300 mL), saturated aqueous solution of $NaHCO_3$ (200 mL), dried over anhydrous $MgSO_4$. Concentration under reduced pressure gave 12.4 g of the crude aldehyde **11** (74% yield), which was used for the next step without further purification.

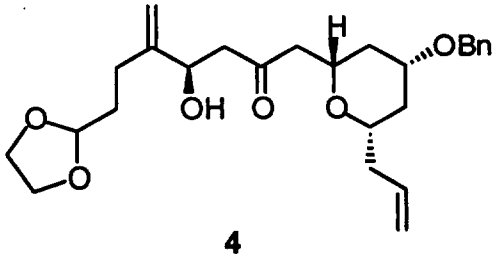


A 100-mL round-bottomed flask, equipped with the Dean-Stark trap was charged with aldehyde **11** (4.66 g, 28.6 mmol), ethylene glycol (1.86 g, 30 mmol), TsOH (50 mg) and benzene (50 mL). The resulting mixture was heated under a gentle reflux for 5 h, partitioned between ether (30 mL) and saturated aqueous solution of $NaHCO_3$ (50 mL). The aqueous layer was extracted with ether (30 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. The resulting dark-brown oil was subjected to the bulb-to-bulb distillation (100 °C oven temperature, 2 mm Hg) to give 5.62 g (96% yield) of the corresponding acetal, as a pale-yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.91 (m, 2H), 2.54 (m, 2H), 3.84 (m, 2H), 3.95 (m, 2H), 4.88 (t, 1H, J = 4.5 Hz), 5.38 (d, 1H, J = 1.7 Hz), 5.59 (q, 1H, J = 1.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 32.3, 35.8, 65.0, 103.1, 116.8, 133.6; IR (neat) 2957, 2881, 1629, 1406, 1138, 1037, 886 cm^{-1}.



5

Aldehyde 5. A solution of vinyl bromide (3.70 g, 17.8 mmol), prepared in the previous step, in THF (50 mL) was treated dropwise with *n*-butyllithium (7.2 mL, 2.5 M in hexane) at –78 °C, followed by addition of dimethylformamide (6.9 mL) after a 20 min period. The reaction mixture was allowed to warm to ambient temperature, quenched by addition of water (200 mL), extracted with ether (2x300 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ether:hexane; 1:3, 1:1) afforded 0.81 g of starting bromide and 1.15 g of aldehyde **5** (53% yield) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.80 (m, 2H), 2.36 (t, 2H, J = 8.0 Hz), 3.83 (m, 2H), 3.94 (m, 2H), 4.86 (t, 1H, J = 4.5 Hz), 6.00 (s, 1H), 6.26 (s, 1H), 9.52 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.3, 31.7, 64.9, 103.7, 134.0, 149.5, 194.4; IR (neat) 2952, 2884, 1688, 1138, 1035, 945 cm^{-1}.

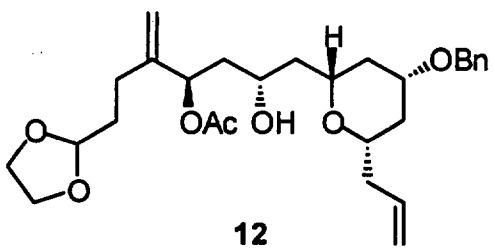


4

Ketone 4 Dicyclohexylboron chloride (2.5 mL of 1.0 M solution in hexane, 2.5 mmol) in ether (20 mL) was cooled to 0 °C, and treated with triethyl amine (0.39 mL, 2.8 mL), followed by addition of ketone **6** (473 mg, 1.64 mmol) in ether (6 mL). The resulting white suspension was stirred for 15 min at 0 °C, cooled to –78 C, and treated with aldehyde **5** (442 mg, 2.83

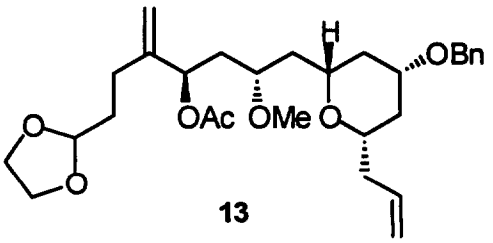
mmol) in ether (6 mL) over a 10 min period. The reaction mixture was stirred for 5 h at –78 C, quenched by addition of 21 mL of methanol:pH 7 buffer (6:1). Upon warming to ambient temperature, 9 mL of methanol-30% hydrogen peroxide (2:1) was added. The stirring was continued for 4 h. The resulting colorless solution was partitioned between ethyl acetate (100 mL) and saturated aqueous solution of $NaHCO_3$ (50 mL). The organic layer was washed with 10% solution of Na_2SO_3, dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:1) afforded 554 mg of ketone **4** (76% yield) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.20 (m, 3H), 1.84 (m, 2H), 2.05 (m, 2H), 2.06-2.30 (m, 4H), 2.43 (dd, 1H, *J* = 15.0, 4.5 Hz), 2.66 (dd, 1H, *J* = 17.0, 9.0 Hz), 2.75 (m, 2H), 3.33 (m, 1H), 3.55 (dddd, 1H, *J* = 11.0, 11.0, 5.0, 5.0 Hz), 3.75 (m, 1H), 3.83 (, 2H), 3.94 (m, 2H), 4.52 (m, 1H), 4.53 (s, 2H), 4.88 (m, 2H), 5.01 (d, 1H, *J* = 9 Hz), 5.03 (d, 1H, *J* = 16.5 Hz), 5.08 (s, 1H), 5.75 (dddd, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz); 7.26-7.34 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 32.1, 37.3, 37.8, 40.3, 49.4, 49.7, 64.9, 69.7, 70.3, 72.2, 74.2, 75.2, 104.0, 110.1, 117.1, 127.6, 128.4, 134.3, 138.4, 149.2, 209.8; IR (neat) 3451, 2916, 2849, 1710, 1352, 1069, 906, 733, 697 cm^{-1}.

Alcohol 12. A solution of ketone **4** (120 mg, 0.27 mmol) and acetaldehyde (48 mg, 1.1 mmol) in THF (3 mL) was cooled to –10 °C and treated with SmI_2 (0.80 mL, 0.1 M solution in THF). The reaction mixture was stirred for 15 min, quenched by addition of saturated aqueous solution of $NaHCO_3$ (15 mL), extracted with ethyl acetate (50 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 2:1) afforded 122 mg of alcohol **12** (93% yield) as a colorless oil. ^{1}H NMR (500 MHz, C_6D_6) δ 1.15 (m, 3H), 1.60 (dt, 1H, *J* = 14.0, 9.5 Hz), 1.72 (s, 3H), 1.65-1.85 (m, 4H), 1.95 (m, 1H), 2.00-2.10 (m, 3H), 2.40 (m, 2H), 2.95 (m, 1H), 3.18 (m, 2H), 3.33 (m, 1H), 3.35 (m, 2H), 3.52 (m, 2H), 3.77 (s, 1H), 3.95 (t, 1H, *J* = 9.0 Hz), 4.34 (d, 1H, *J* = 12.5 Hz), 4.36 (d, 1H, *J* = 12.5 Hz), 4.84 (t, 1H, *J* = 4.5 Hz), 4.89 (s, 1H), 4.98 (m, 2H), 5.15 (s, 1H), 5.66 (dddd, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz); 5.87 (dd, 1H, *J* = 9.5, 2.5 Hz), 7.13 (t, *J* = 7.0 Hz), 7.22 (t, 2H, *J* = 7.0 Hz), 7.34 (d, 2H, *J* = 7 Hz); ^{13}C NMR (125 MHz, C_6D_6) δ 20.7, 26.9, 32.7, 37.8, 38.8, 40.7, 42.6, 43.3, 64.8, 67.7, 69.4, 73.8, 74.3, 75.2, 76.5, 104.2, 110.4, 117.5, 127.5, 127.6, 128.6, 134,7, 139.6, 148.9, 169.6; IR (neat) 3492, 2917, 2860, 1736, 1642, 1368, 1236, 1070, 904, 736, 698 cm^{-1}.



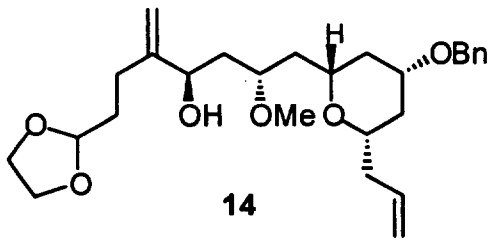
12

Methyl Ether 13. A solution of alcohol **12** (110 mg, 0.225 mmol), 2,6-di-*tert*-butylpyridine (0.76 mL, 3.37 mmol) and methyl triflate (0.323 mL, 2.25 mmol) was stirred for 14 h at ambient temperature, and quenched by addition of aqueous solution of ammonium hydroxide (10 mL). The product was extracted with ether (2x50 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. The excess 2,6-di-*tert*-butylpyridine was removed by bulb-to-bulb distillation (80 °C, 0.5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 81 mg of methyl ether **13** (72% yield) as a colorless oil. ^{1}H NMR (500 MHz, C_6D_6) δ 1.19 (q, 1H, J = 11.5 Hz), 1.26 (q, 1H, J = 11.5 Hz), 1.43 (ddd, 1H, J = 14.0, 7.5, 3.5 Hz), 1.77 (s, 3H), 1.80-1.90 (m, 5H), 2.00-2.10 (m, 3H), 2.27 (m, 1H), 2.40 (m, 2H), 2.98 (m, 1H), 3.17 (s, 3H), 3.20-3.28 (m, 2H), 3.33 (m, 1H), 3.35 (m, 2H), 3.50 (m, 2H), 4.37 (s, 2H), 4.83 (t, 1H, J = 4.5 Hz), 4.89 (s, 1H), 5.03 (m, 2H), 5.17 (s, 1H), 5.84 (dd, 1H, J = 9.5, 2.5 Hz), 5.90 (m, 1H); 7.12 (t, J = 7.0 Hz), 7.22 (t, 2H, J = 7.0 Hz), 7. 35 (d, 2H, J = 7 Hz); ^{13}C NMR (125 MHz, C_6D_6) δ 20.7, 26.8, 32.7, 38.1, 39.0, 39.4, 39.8, 40.9, 56.5, 64.8, 69.4, 72.1, 73.8, 74.7, 74.8, 75.2, 104.2, 110.5, 116.6, 127.5, 127.6, 128.5, 135,5, 139.7, 148.8, 169.3; IR (neat) 2939, 2880, 1736, 1368, 1235, 1070, 906, 736, 698 cm^{-1}.



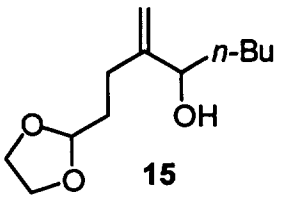
13

Alcohol 14. Lithium aluminum hydride (38 mg, 1 mmol) was suspended in ether (2 mL), cooled to –78 °C, and treated with methyl ether **13** (66 mg, 0.13 mmol) in ether (2 mL). The reaction mixture was stirred for 30 min at –78 °C, quenched by dropwise addition of water (0.2 mL). The resulting suspension was allowed to reach ambient temperature, vigorously stirred for 1 h, diluted with ether, dried with anhydrous Na_2SO_4, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 51 mg of alcohol **14** (86% yield) as a colorless oil. ^{1}H NMR (500 MHz, C_6D_6) δ 1.20 (q, 1H, J = 11.5 Hz), 1.26 (q, 1H, J = 11.5 Hz), 1.46 (ddd, 1H, J = 14.0, 7.5, 3.5 Hz), 1.80-1.85 (m, 4H), 1.93 (ddd, 1H, J = 14.0, 9.0, 4.5 Hz), 2.00-2.04 (m, 2H), 2.10 (m, 1H), 2.28 (m, 2H), 2.40 (m, 1H), 3.00 (m, 1H), 3.08 (s, 3H), 3.20-3.28 (m, 2H), 3.35 (m, 2H), 3.69 (m, 1H), 4.37 (s, 2H), 4.43 (m, 1H), 4.85 (t, 1H, J = 4.5 Hz), 4.95 (s, 1H), 5.04 (m, 2H), 5.33 (s, 1H), 5.86 (dddd, 1H, J = 17.0, 10.0, 7.0, 7.0 Hz), 7.12 (t, J = 7.0 Hz), 7.22 (t, 2H, J = 7.0 Hz), 7. 35 (d, 2H, J = 7 Hz); ^{13}C NMR (125 MHz, C_6D_6) δ 26.7, 33.0, 37.9, 39.0, 38.9, 39.3, 39.5, 40.9, 56.0, 64.8, 69.4, 72.3, 74.8, 75.2, 76.4, 104.4, 108.9, 116.8, 127.5, 127.6, 128.5, 135,3, 139.7, 152.2; IR (neat) 3452, 2942, 2878, 1642, 1354, 1071, 904, 736, 698 cm^{-1}.



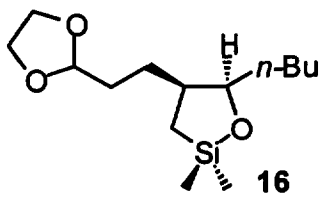
14

Alcohol 15. Aldehyde **5** (312 mg, 2.0 mmol) in THF (10 mL) was cooled to –78 °C, and treated with *n*-butyllithium (0.88 mL, 2.5 M solution in hexane). After 15 min, the reaction was quenched by addition of saturated aqueous solution of $NaHCO_3$ (15 mL), extracted with ether (50 mL), dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ether:hexane; 2:1) afforded 380 mg of alcohol **15** (87% yield) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (t, 3H, *J* = 7.0 Hz), 1.20-1.38 (m, 4H), 1.53 (m, 2H), 1.78 (br s, 1H), 1.85 (m, 2H), 2.10 (m, 1H), 2.21 (m, 1H), 3.81 (m, 2H), 3.95 (m, 2H), 4.06 (t, 1H, *J* = 7.0 Hz), 4.83 (s, 1H), 4.87 (t, 1H, *J* = 4.5 Hz), 5.01 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 22.6, 25.3, 27.8, 32.2, 35.1, 64.9, 75.6, 104.2, 109.8, 151.1; IR (neat) 3439, 2953, 2870, 1692, 1409, 1135, 1031, 899, 731 cm^{-1}.



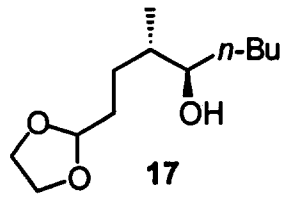
15

Silane 16. A solution of alcohol **15** (142 mg, 0.66 mmol) in CH_2Cl_2 (0.50 mL) was treated with tetramethyldisilazane (0.35 mL, 2.0 mmol). After 12 h, ^{1}H NMR analysis indicated complete consumption of the starting alcohol. The excess tetramethyldisilazane was removed under high vacuum. The resulting silyl ether was dissolved in C_6H_6 (7 mL) and treated with H_2PtCl_6 [30 μL, 0.05 M in THF, (0.3 mol%)]. After 30 min at 50 °C, ^{1}H NMR analysis indicated quantitative formation of the silacycle **16**, as 85:15 mixture of diastereomers. Major isomer: ^{1}H NMR (400 MHz, C_6D_6) δ 0.10 (s, 3H), 0.15 (s, 3H), 0.90 (t, 3H, *J* = 7.0 Hz), 1.20-1.90 (m, 10H), 2.00 (m, 1H), 3.39 (m, 2H), 3.56 (m, 2H), 3.99 (ddd, 1H, *J* = 10.4, 5.6, 3.2 Hz), 4.82 (t, 1H, *J* = 4.5 Hz); ^{13}C NMR (100 MHz, C_6D_6) δ 0.2, 1.2, 14.4, 16.2, 23.0, 26.9, 28.7, 31.4, 33.2, 42.5, 64.8, 104.8; IR (neat) 2953, 2871, 1408, 1250, 1033, 840, 796 cm^{-1}.



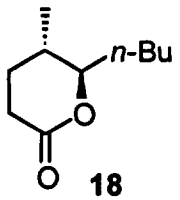
16

Alcohol 17. Cyclic silane **16**, obtained upon concentration of the crude hydrosilylation reaction mixture was treated with TBAF (2.6 mmol) in DMF (2.5 mL). The resulting solution was heated to 75 °C for 24 h. The excess DMF was removed by bulb-to-bulb distillation (50 °C, 0.5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 121 mg of alcohol **17** (82% yield from **15**) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.87 (d, 3H, *J* = 7.0 Hz), 0.88 (t, 3H, *J* = 7.0 Hz), 1.20-1.90 (m, 10H), 2.74 (m, 1H), 3.41 (m, 1H), 3.82 (m, 2H), 3.95 (m, 2H), 4.83 (t, 1H, *J* = 4.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 15.2, 22.7, 26.0, 28.2, 31.6, 33.1, 38.6, 64.8, 75.7, 104.7.



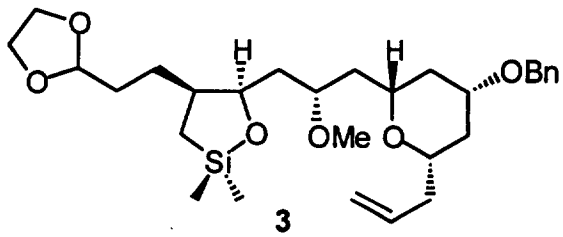
17

Lactone 18. A solution of alcohol **17** (110 mg, .50 mmol) in 6 mL of THF-water (5:1) containing a drop of concentrated H_2SO_4 was heated to 80 °C for 10 h, then partitioned between saturated aqueous solution of $NaHCO_3$ (1 mL), and ether (20 mL). The organic layer was separated, dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ether:pentane; 1:1) afforded 62 mg of the corresponding lactol as a colorless oil. The oxidation was conducted by addition of bromine (15 μL) to the solution of the lactol in acetic acid (0.4 mL) and water (0.64 mL). After 1 h, the reaction mixture was partitioned between saturated aqueous solution of $NaHCO_3$, and ether (20 mL). The organic layer was separated, dried over anhydrous $MgSO_4$, filtered and concentrated. Flash chromatography on silica gel (elution with ether:pentane; 1:1) afforded 61 mg of lactone **18** (86% from 17) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, 3H, J = 7.0 Hz), 0.97 (t, 3H, J = 7.0 Hz), 1.20-1.70 (m, 8H), 1.87 (m, 1H), 2.45 (ddd, 1H, J = 17.0, 10.0, 6.8 Hz), 2.60 (ddd, 1H, J = 17.0, 6.4, 4.4 Hz), 3.91 (ddd, 1H, J = 10.0, 8.0, 3.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 17.4, 22.6, 26.5, 27.8, 29.5, 32.2, 33.1, 85.9, 174.0.

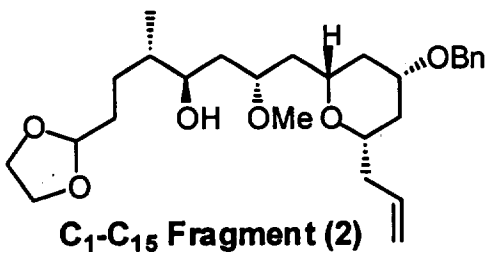


18

Silane 3. A solution of alcohol **13** (46 mg, 0.10 mmol) in $CDCl_3$ (0.50 mL) was treated with tetramethyldisilazane (0.10 mL, 0.56 mmol). After 15 min, ^{1}H NMR analysis indicated complete consumption of the starting alcohol. The excess tetramethyldisilazane was removed under high vacuum. The resulting silyl ether was dissolved in C_6D_6 (1 mL) and treated with H_2PtCl_6 (0.5 mol%). After 10 min at 57 °C, ^{1}H NMR analysis indicated quantitative formation of the silacycle **3**, as 87:13 mixture of diastereomers. Major isomer: ^{1}H NMR (500 MHz, C_6D_6) δ 0.11 (s, 3H), 0.17 (s, 3H), 0.48 (dd, 1H, J = 14.5, 11.0 Hz), 0.80 (dd, 1H, J = 14.5, 7.0 Hz), 1.25 (q, 1H, J = 11.5 Hz), 1.36 (q, 1H, J = 11.5 Hz), 1.46-2.38 (m, 2H), 3.10 (m, 1H), 3.33 (s, 3H), 3.20-3.28 (m, 2H), 3.28 (m, 2H), 3.35 (m, 2H), 3.90 (m, 1H), 4.38 (s, 2H), 4.50 (m, 1H), 4.85 (t, 1H, J = 4.5 Hz), 5.04 (m, 2H), 5.92 (m, 1H), 7.11 (t, J = 7.0 Hz), 7.21 (t, 2H, J = 7.0 Hz), 7. 34 (d, 2H, J = 7 Hz).



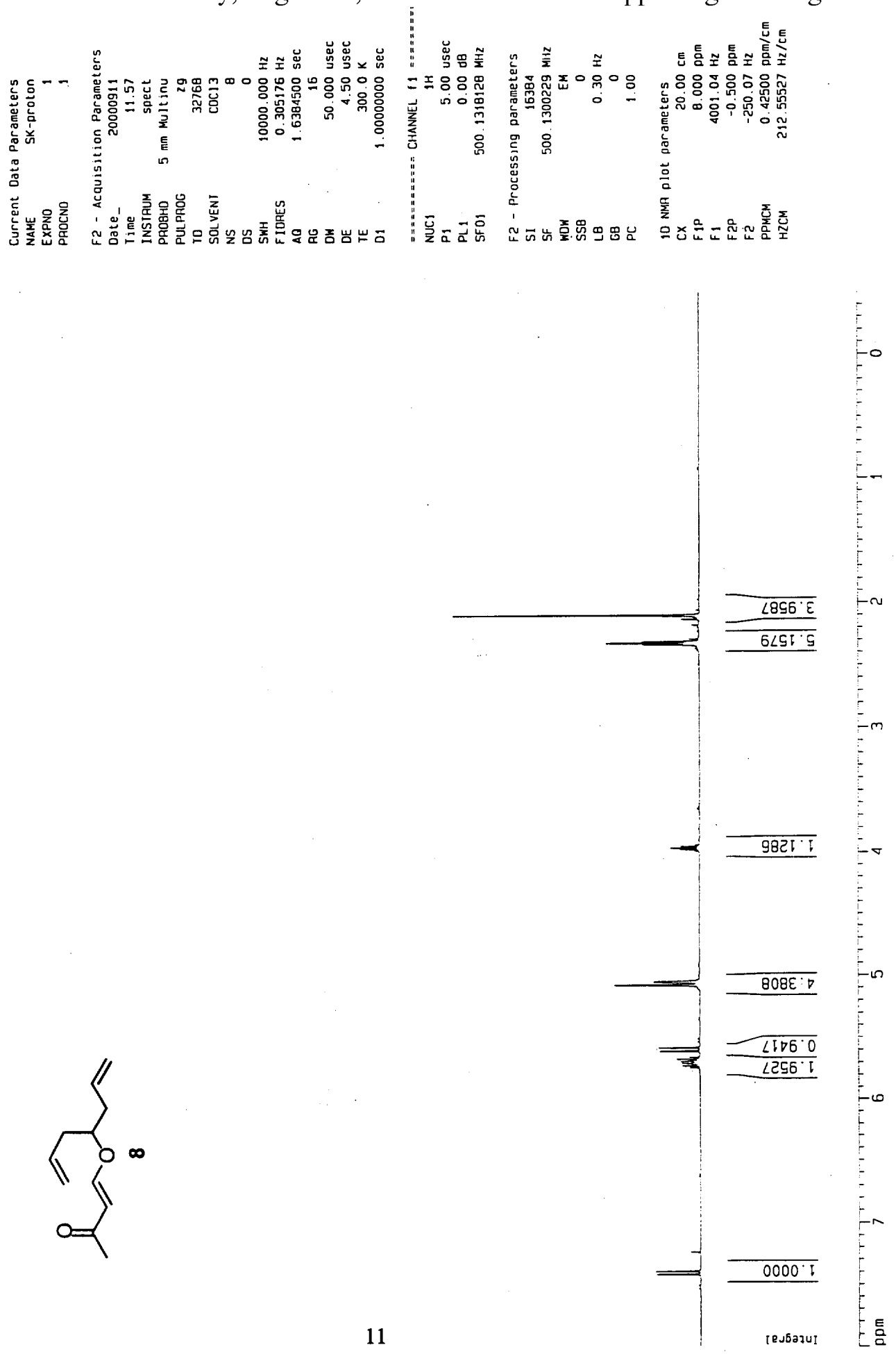
3



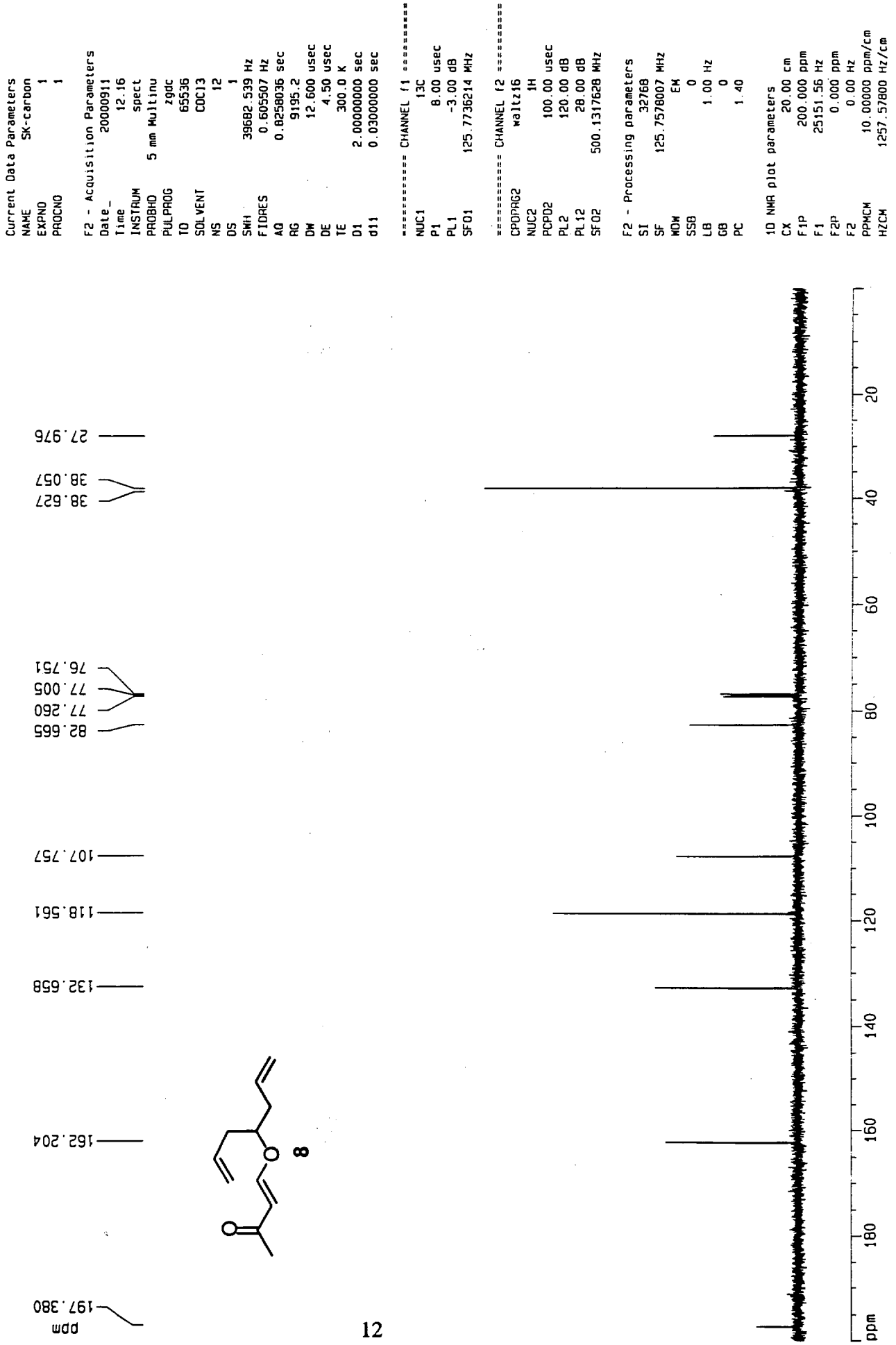
C_1-C_{15} Fragment (2)

Alcohol 2. Cyclic silane **3**, obtained upon concentration of the crude hydrosilylation reaction mixture was treated with TBAF (0.4 mmol) in *d*-DMF (0.5 mL). The resulting solution was heated to 70 °C for 15 min. At this point, ^{1}H NMR analysis indicated complete consumption of the starting silane **3**. The excess DMF was removed by bulb-to-bulb distillation (50 °C, 0. 5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:1) afforded 24 mg of alcohol **2** (54% yield from **14**) as a colorless oil. ^{1}H NMR (500 MHz, C_6D_6) δ 0.93 (d, 3H, *J* = 6.5 Hz), 1.22 (q, 1H, *J* = 11.5 Hz), 1.28 (q, 1H, *J* = 11.5 Hz), 1.48-2.03 (m, H), 2.10 (m, 1H), 2.30 (m, 1H), 2.80 (br s, 1H), 3.00 (m, 1H), 3.07 (s, 3H), 3.26 (m, 2H), 3.36 (m, 2H), 3.56 (m, 2H), 3.67 (m, 1H), 3.78 (m, 1H), 4.37 (s, 2H), 4.85 (t, 1H, *J* = 4.5 Hz), 5.04 (m, 2H), 5.87 (dddd, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz), 7.12 (t, *J* = 7.0 Hz), 7.22 (t, 2H, *J* = 7.0 Hz), 7. 35 (d, 2H, *J* = 7 Hz); ^{13}C NMR (125 MHz, C_6D_6) δ 15.4, 27.0, 32.2, 36.3, 37.9, 38.9, 39.3, 39.4, 40.9, 56.2, 64.8, 69.4, 72.4, 74.9, 75.2, 76.8, 105.2, 116.8, 127.5, 127.6, 128.5, 135,2, 139.7; IR (neat) 3470, 2919, 2870, 1354, 1071, 736, 697 cm^{-1}.

^{1}H NMR and ^{13}C NMR Spectra



8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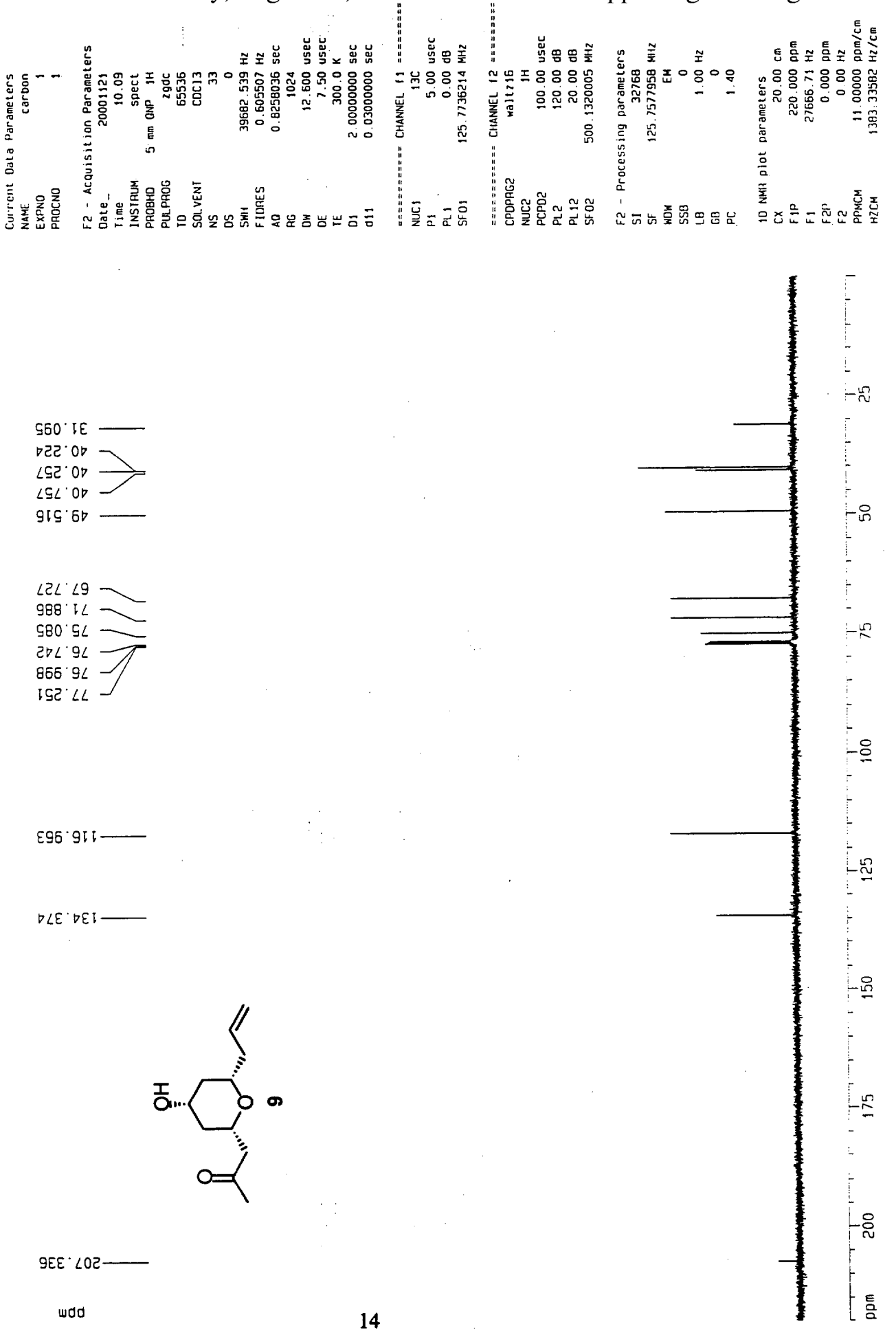
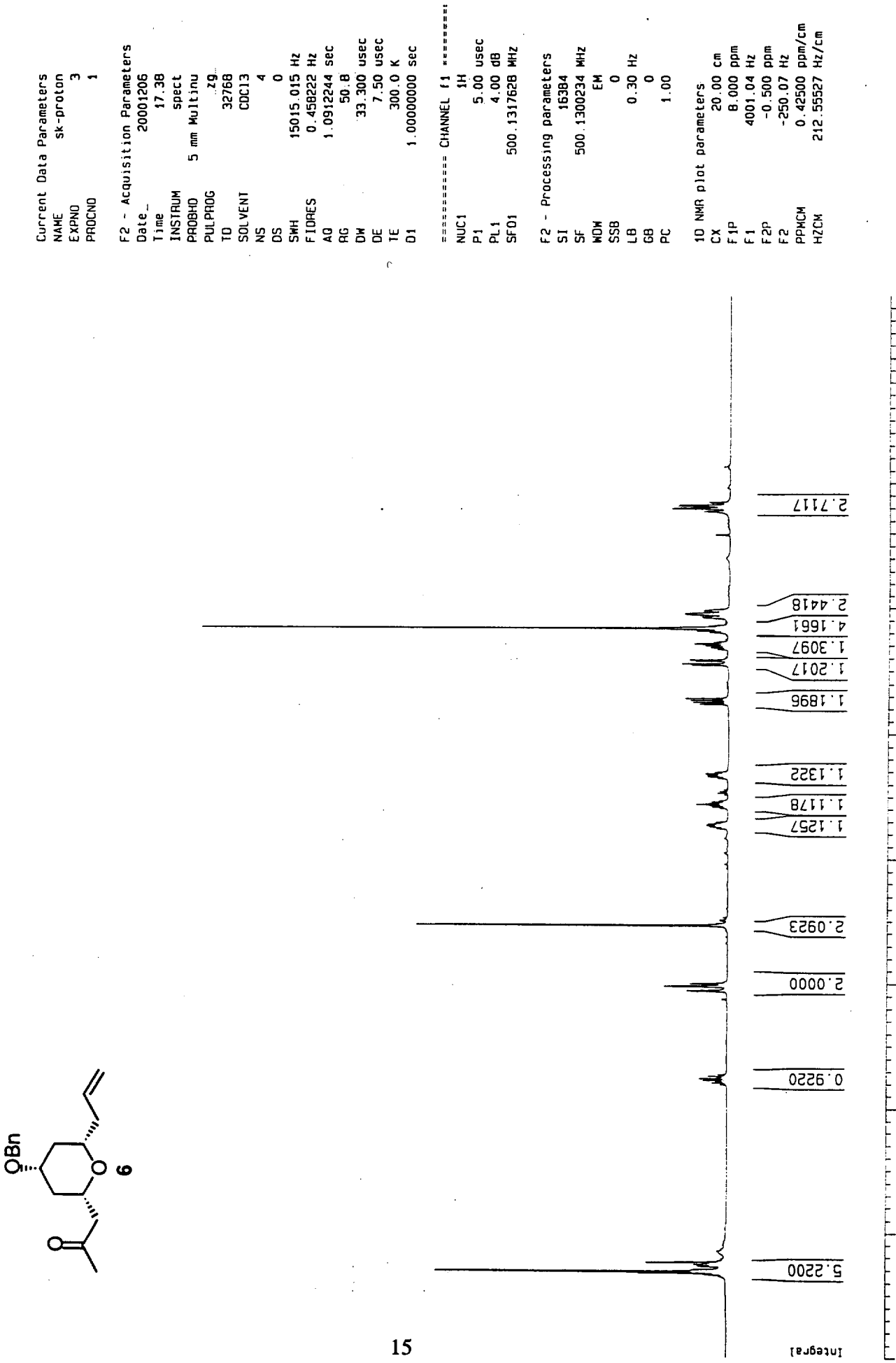
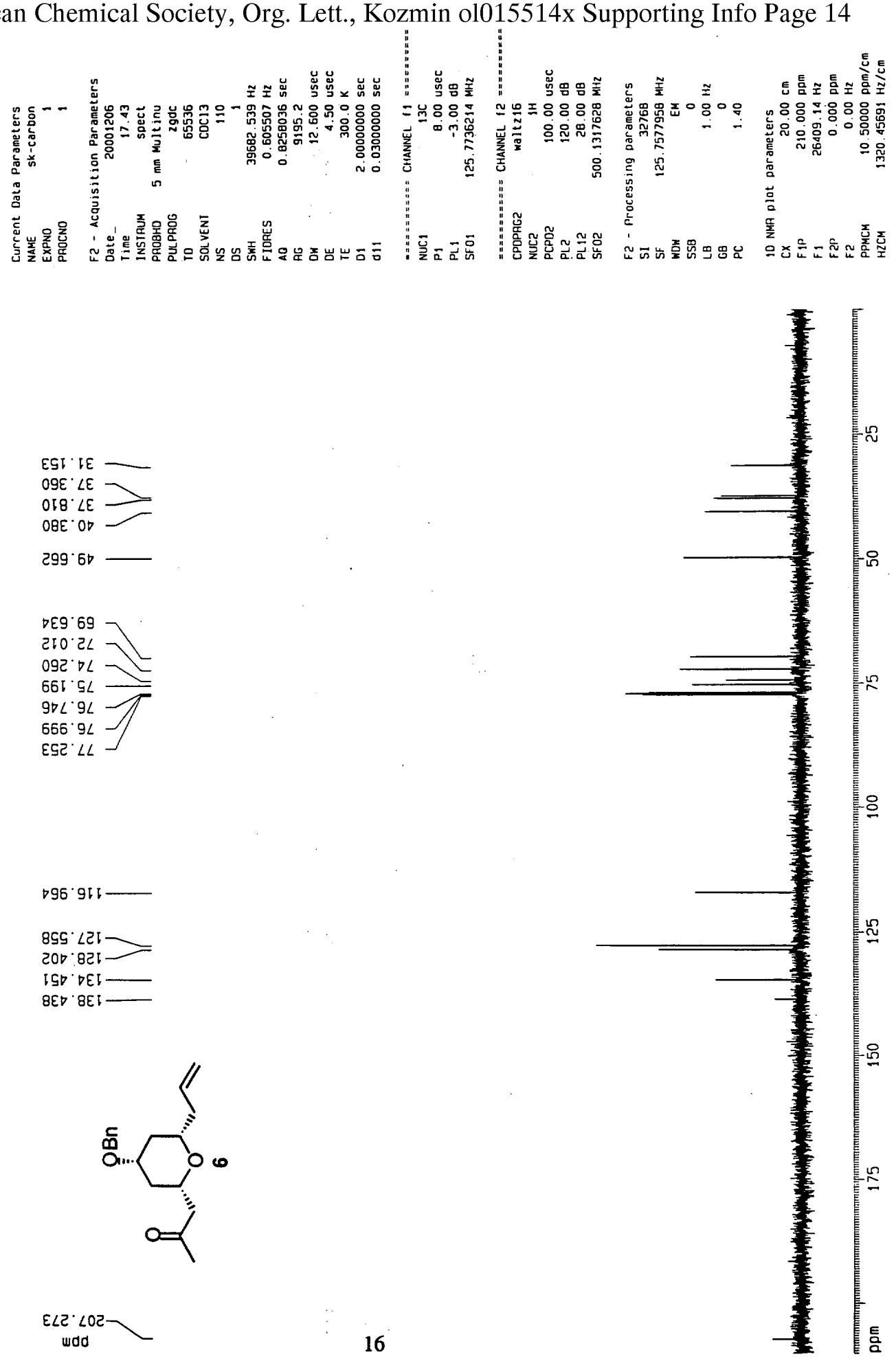
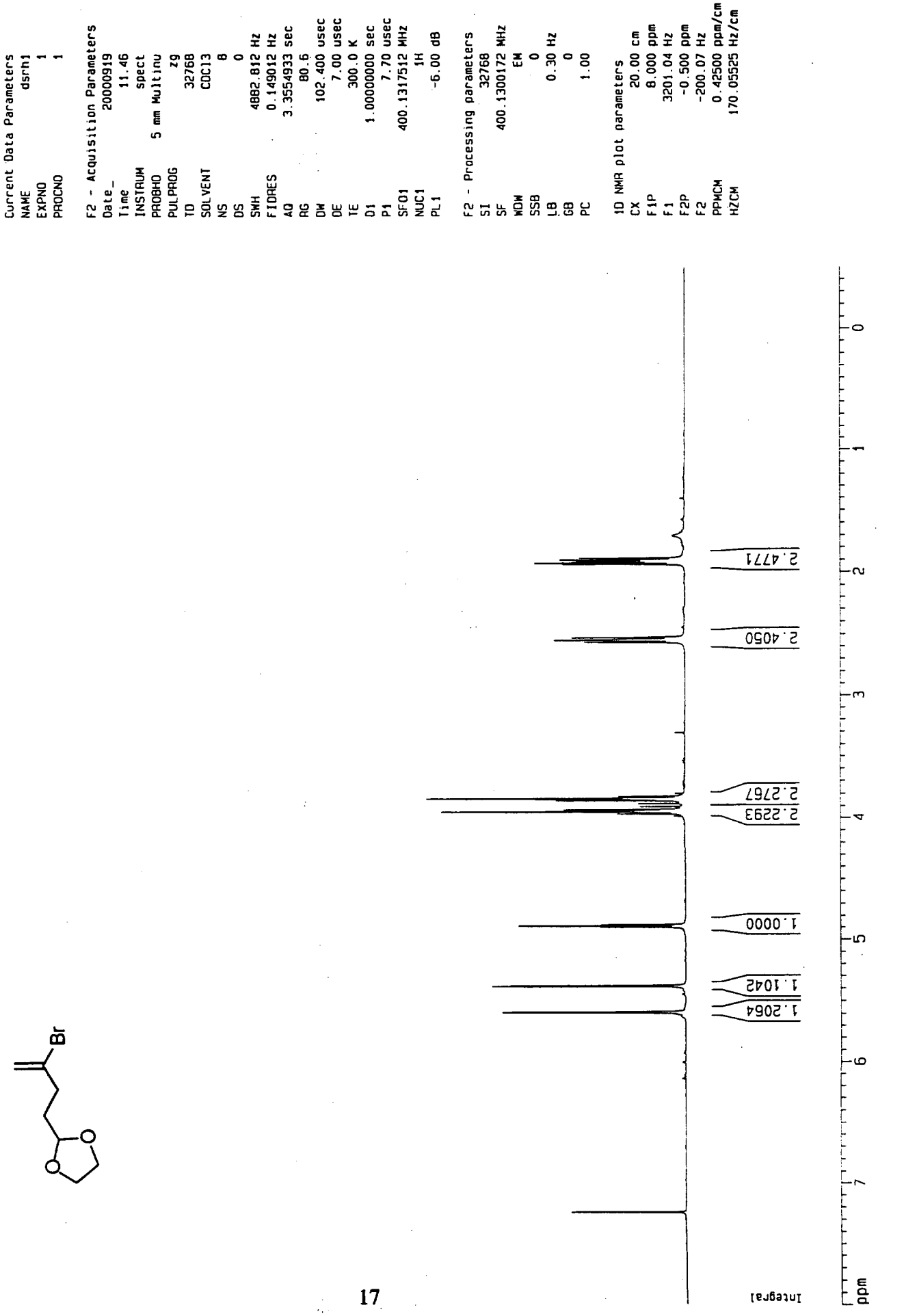
AQ 0.8258036 sec
RG 9195.2
DW 12.600 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 125.7736214 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 28.00 dB
SFO2 500.1317628 MHz
F2 - Processing parameters
SI 32768
SF 125.7578007 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1257.57800 Hz/cm
27.976
38.057
38.627
76.751
77.005
77.260
82.665
107.757
118.561
132.658
162.204
197.380
ppm
8
ppm

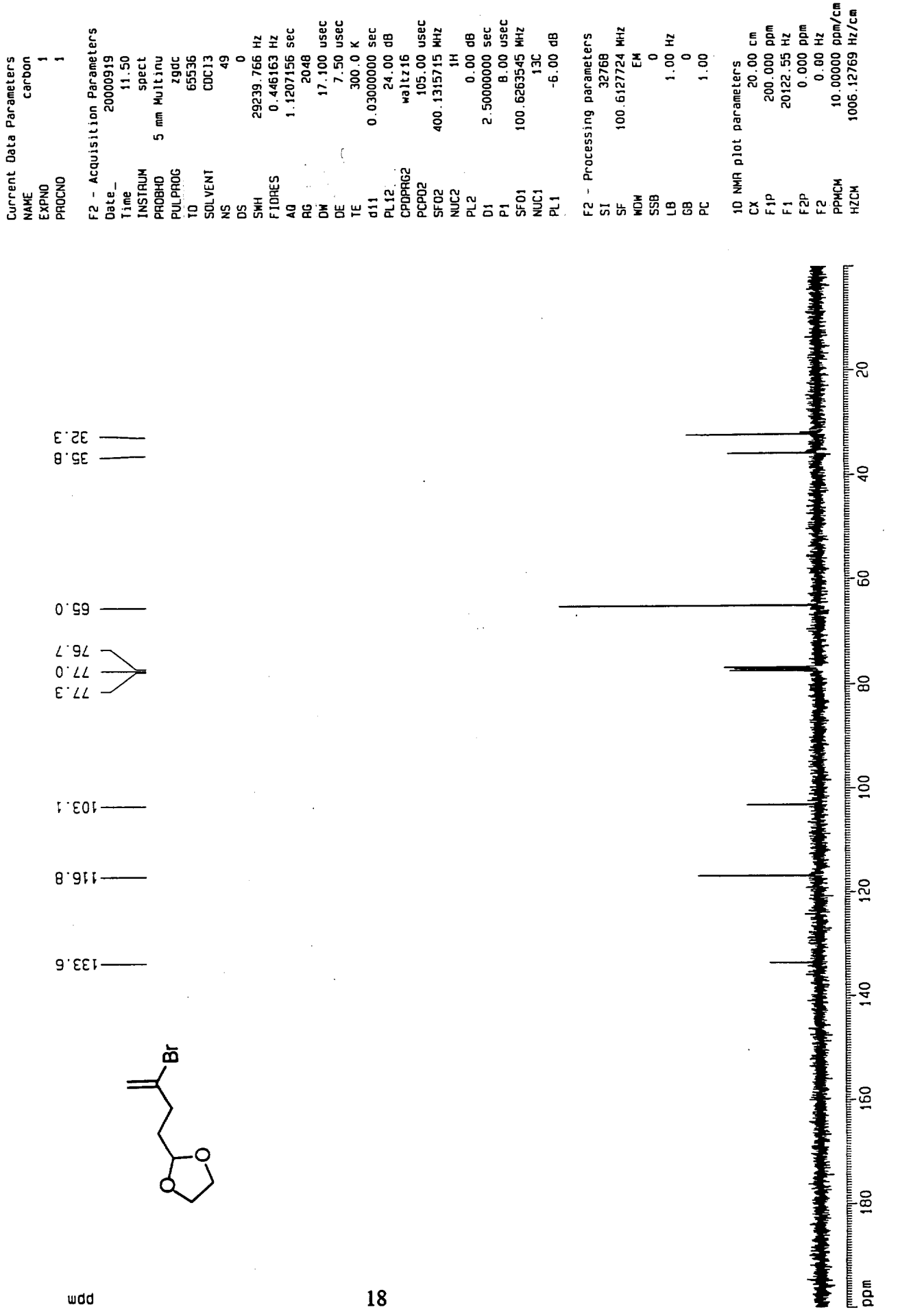

Current Data Parameters
NAME proton
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20001121
Time 10.07
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5208.333 Hz
FIDRES 0.158946 Hz
AQ 3.1457779 sec
RG 64
DW 96.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1320118 MHz
F2 - Processing parameters
SI 16384
SF 500.1300239 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 212.55527 Hz/cm
OH
O
O
9
0.0462
0.0988
0.0952
0.0472
0.0484
0.0524
0.0591
0.1979
0.1202
0.1060
Integral
ppm
0
1
2
3
4
5
6
7

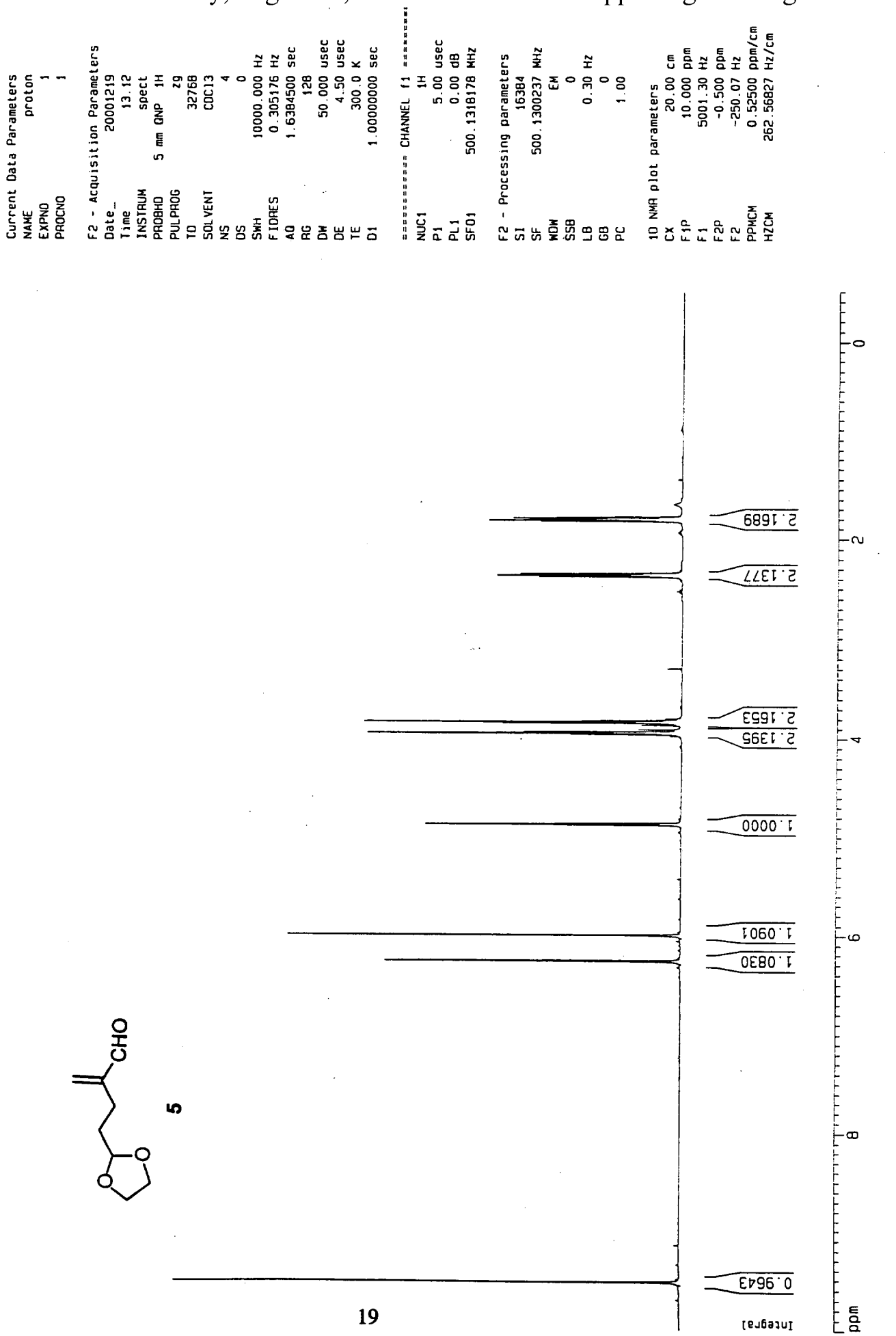


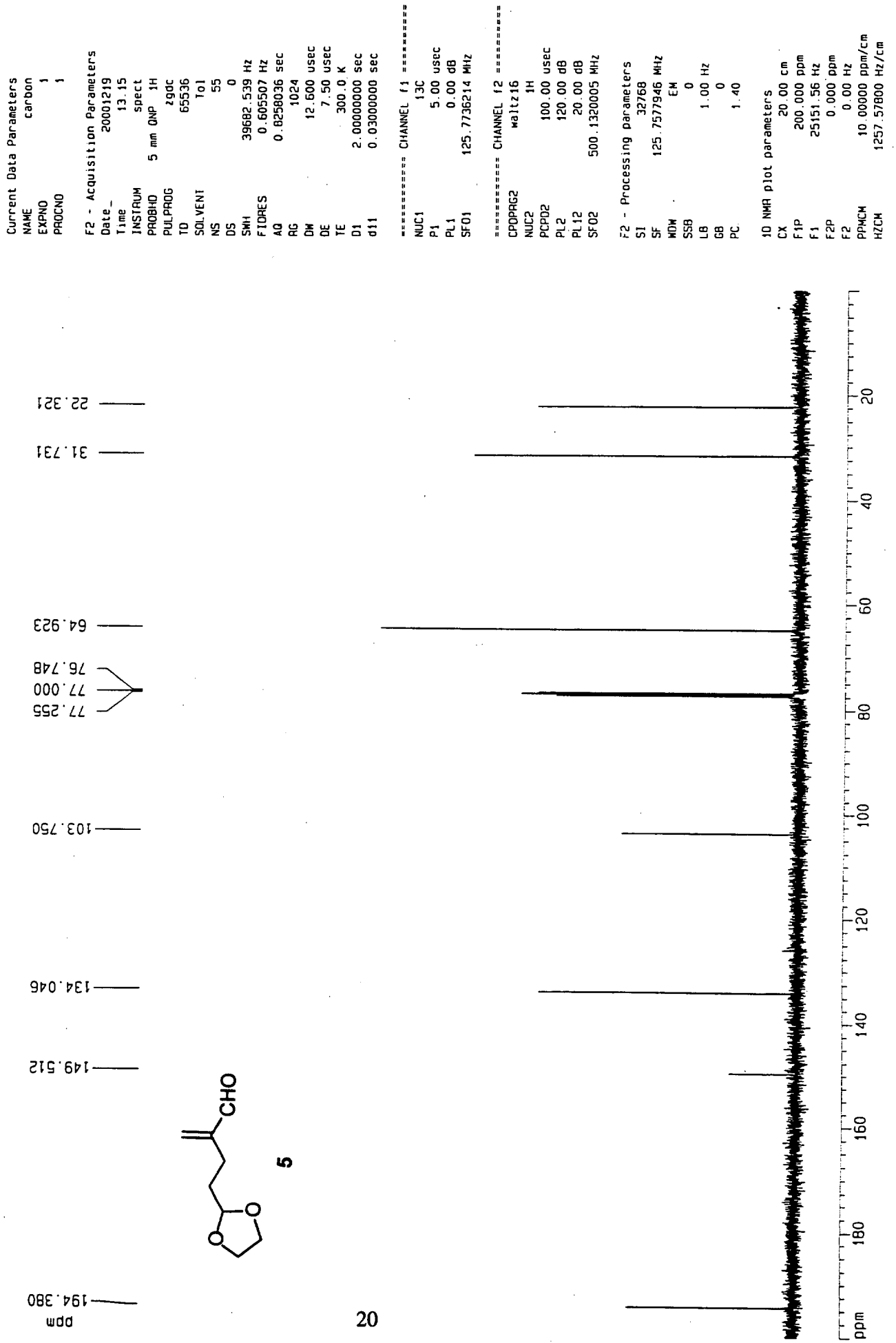


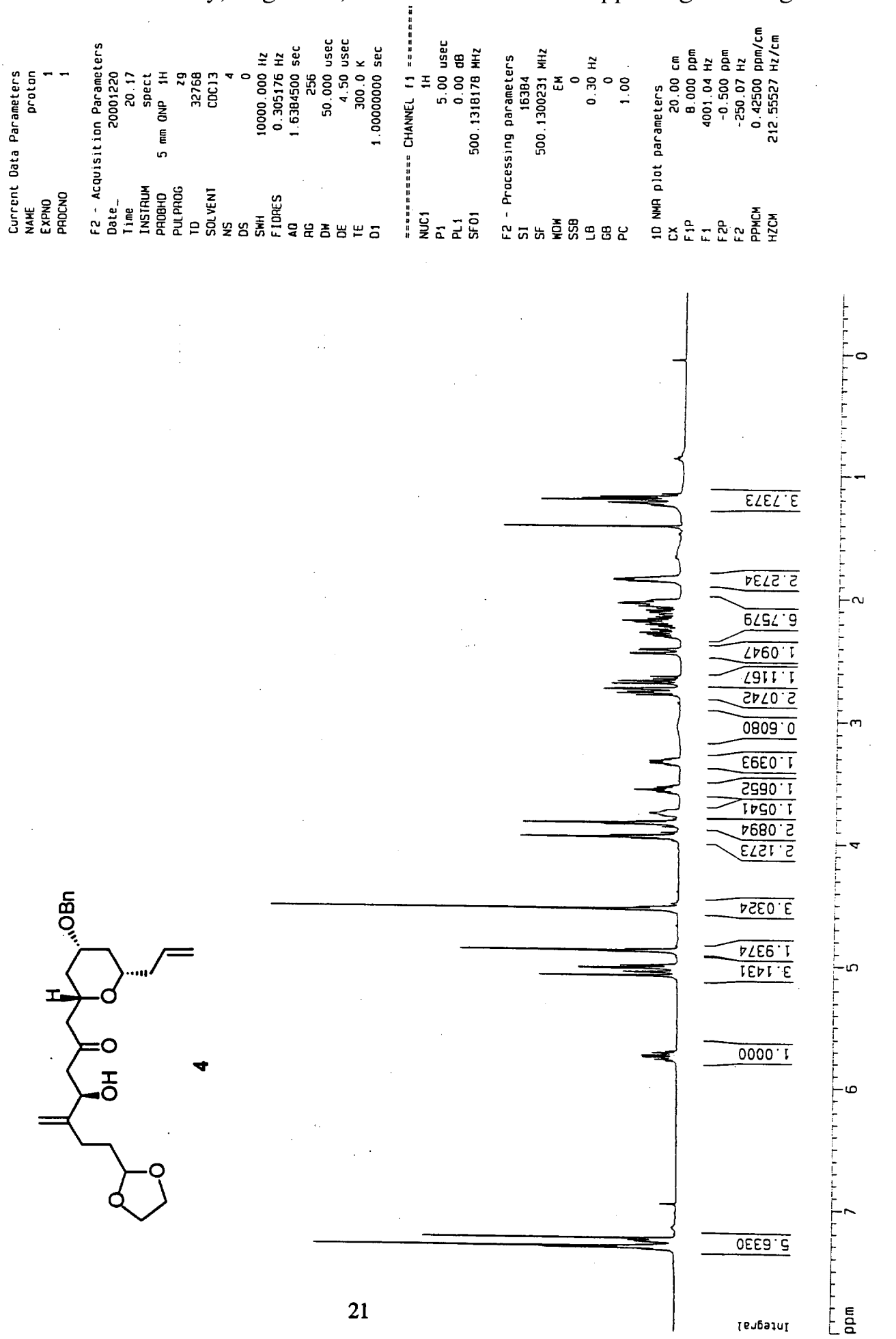








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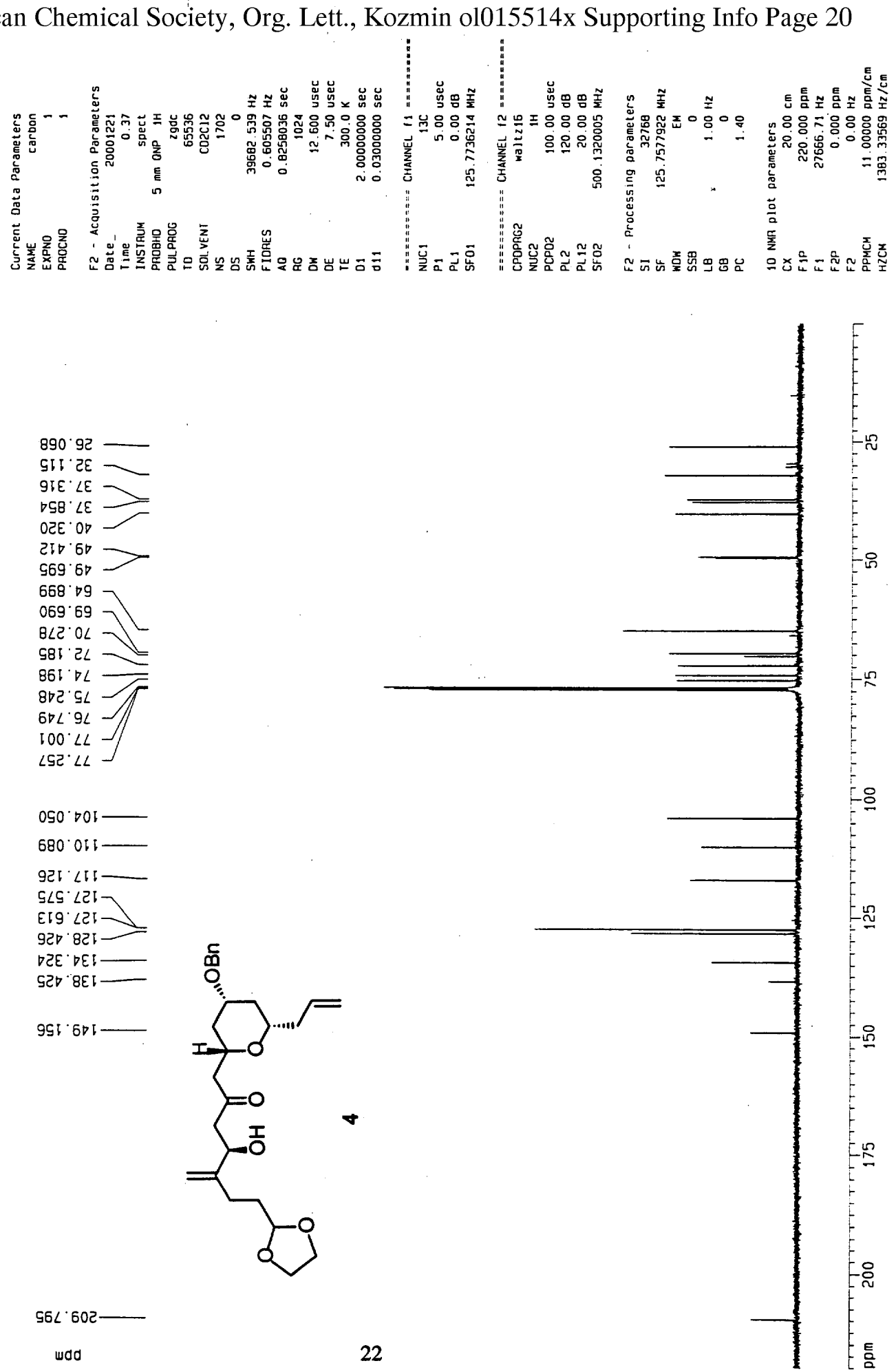
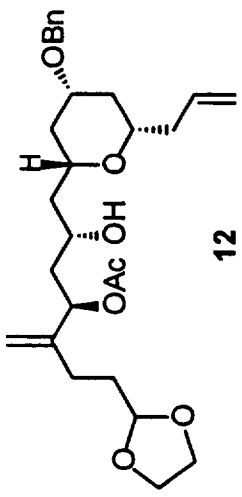
NAME proton
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20001220
Time 20.17
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 256
DW 50.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1318178 MHz
F2 - Processing parameters
SI 16384
SF 500.1300231 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 212.55527 Hz/cm
OBn
H
O
O
OH
O
4
3.7373
2.2734
6.7579
1.0947
1.1167
2.0742
0.6080
1.0393
1.0652
1.0541
2.0894
2.1273
3.0324
1.9374
3.1431
1.0000
5.6330
Integral
ppm





12

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Current Data Parameters
NAME          proton
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_       20001221
Time           18.01
INSTRUM         spect
PROBHD  5 mm QNP 1H
PULPROG            zg
TD              32768
SOLVENT          C6D6
NS                  4
DS                  0
SWH         10000.000 Hz
FIDRES          0.305176 Hz
AQ              1.6384500 sec
RG                 32
DW             50.000 usec
DE              4.50 usec
TE              300.0 K
D1              1.00000000 sec
========== CHANNEL f1 ==========
NUC1               1H
P1               5.00 usec
PL1              0.00 dB
SFO1        500.1318178 MHz

F2 - Processing parameters
SI              16384
SF         500.1300620 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC              1.00

1D NMR plot parameters
CX             20.00 cm
F1P             8.000 ppm
F1           4001.04 Hz
F2P            -0.500 ppm
F2           -250.07 Hz
PPMCM          0.42500 ppm/cm
HZCM         212.55528 Hz/cm
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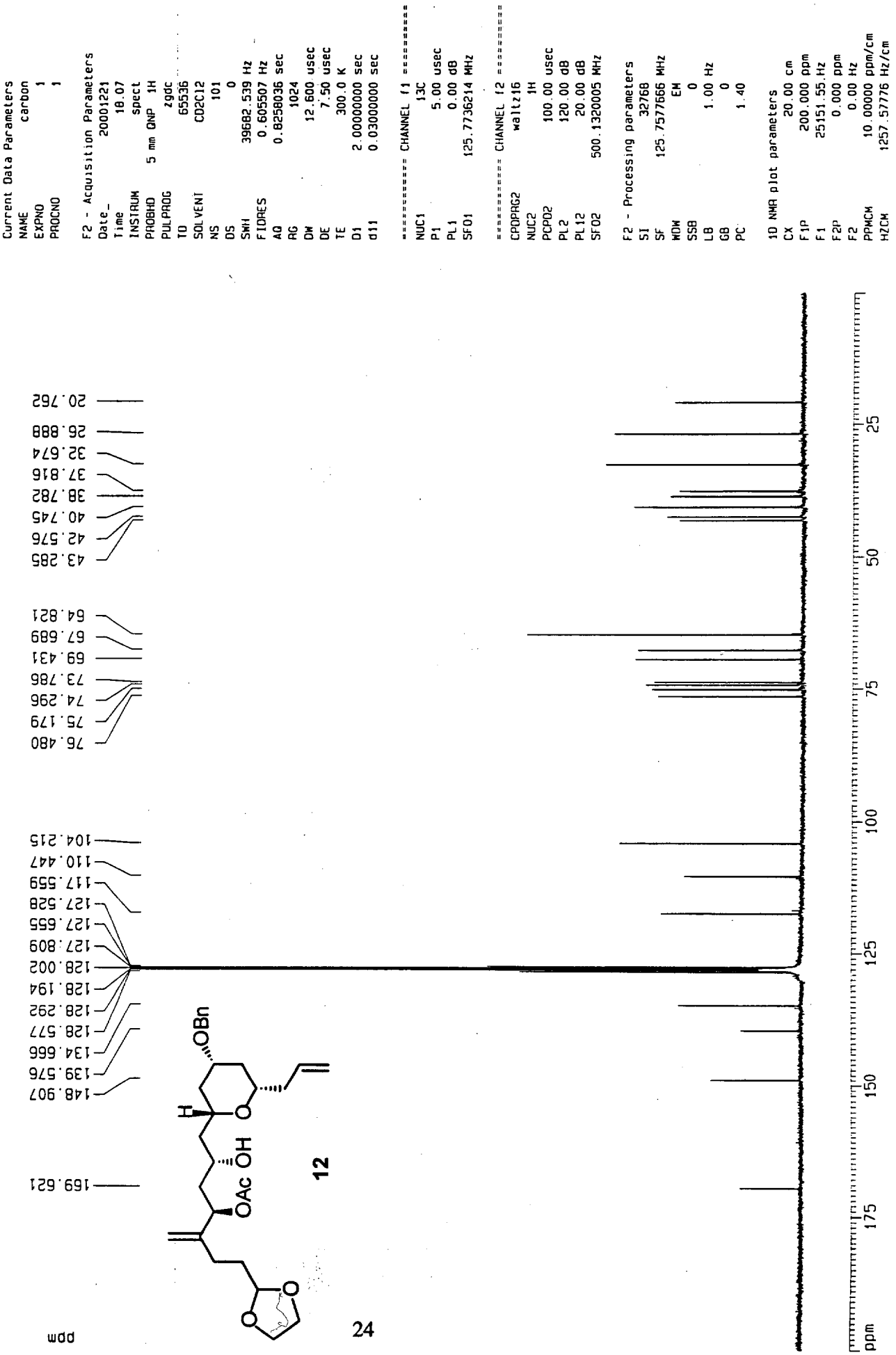

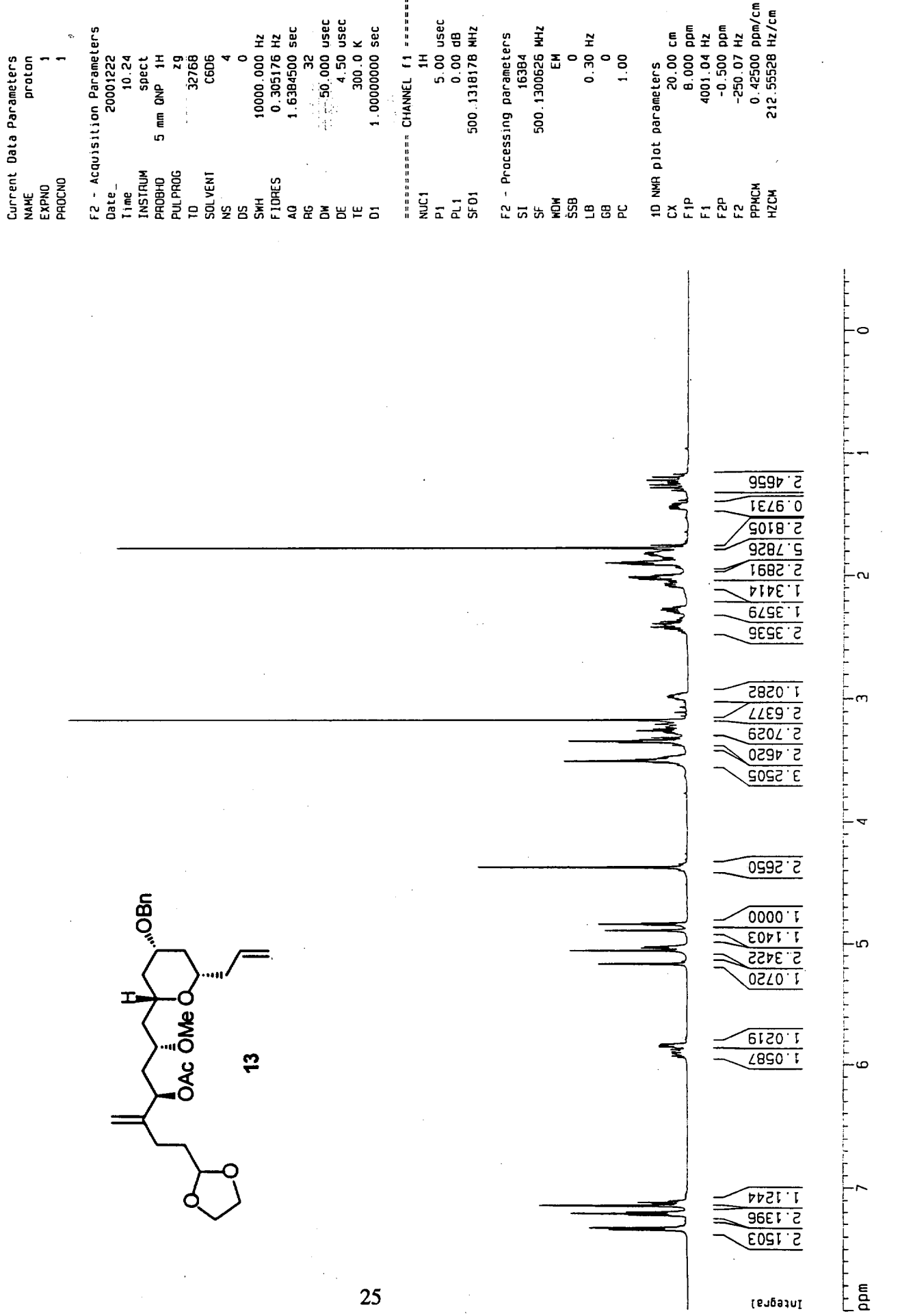

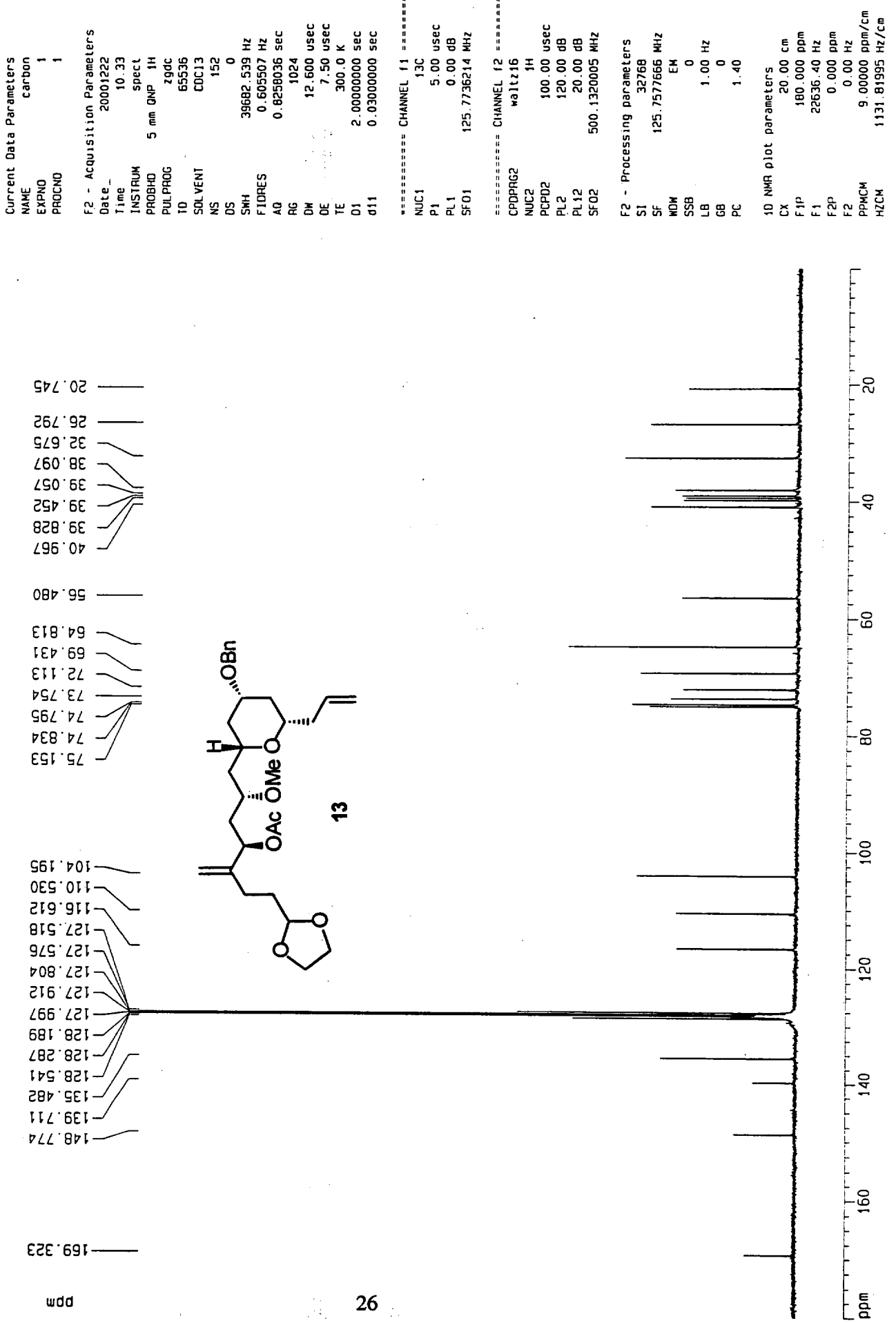

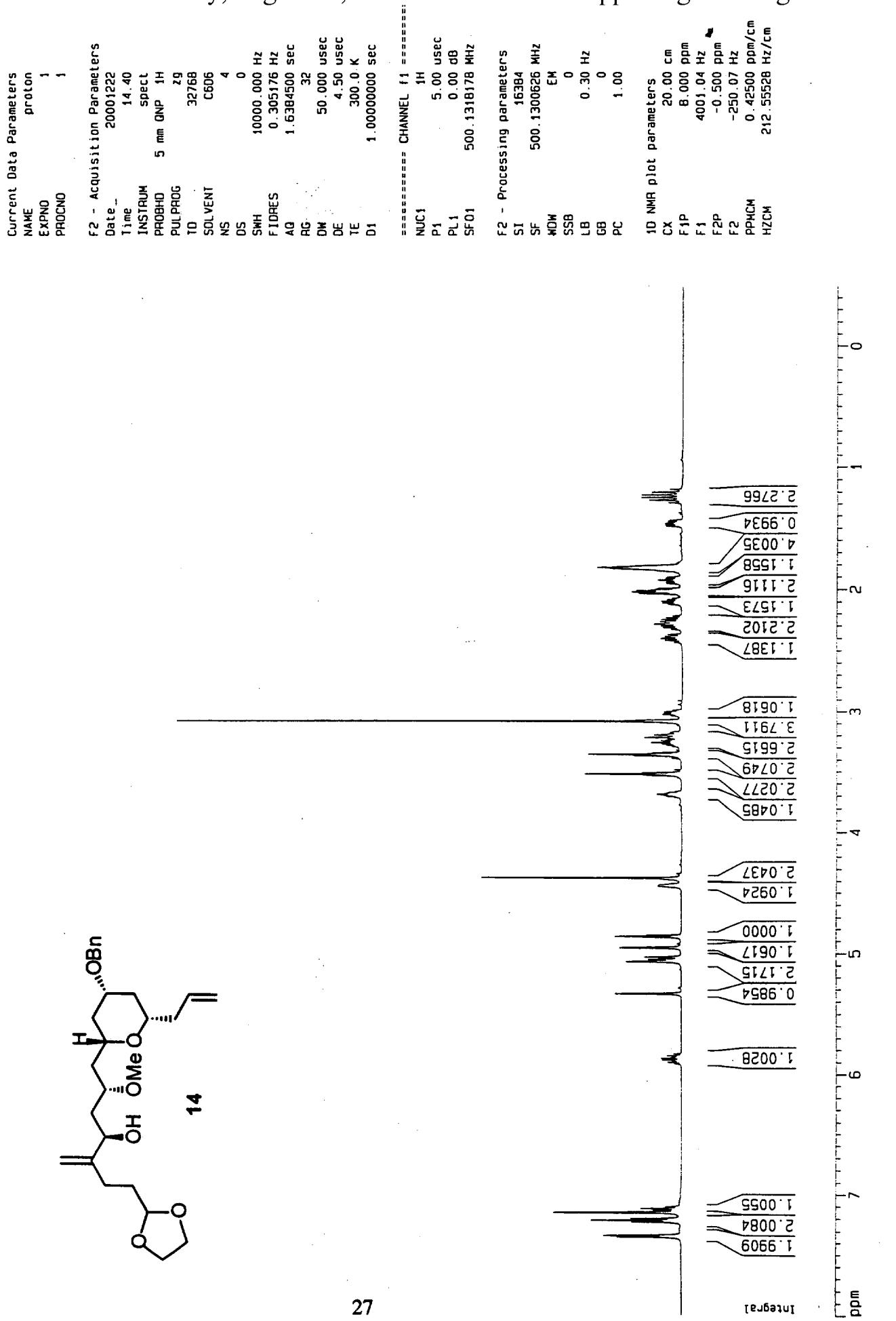

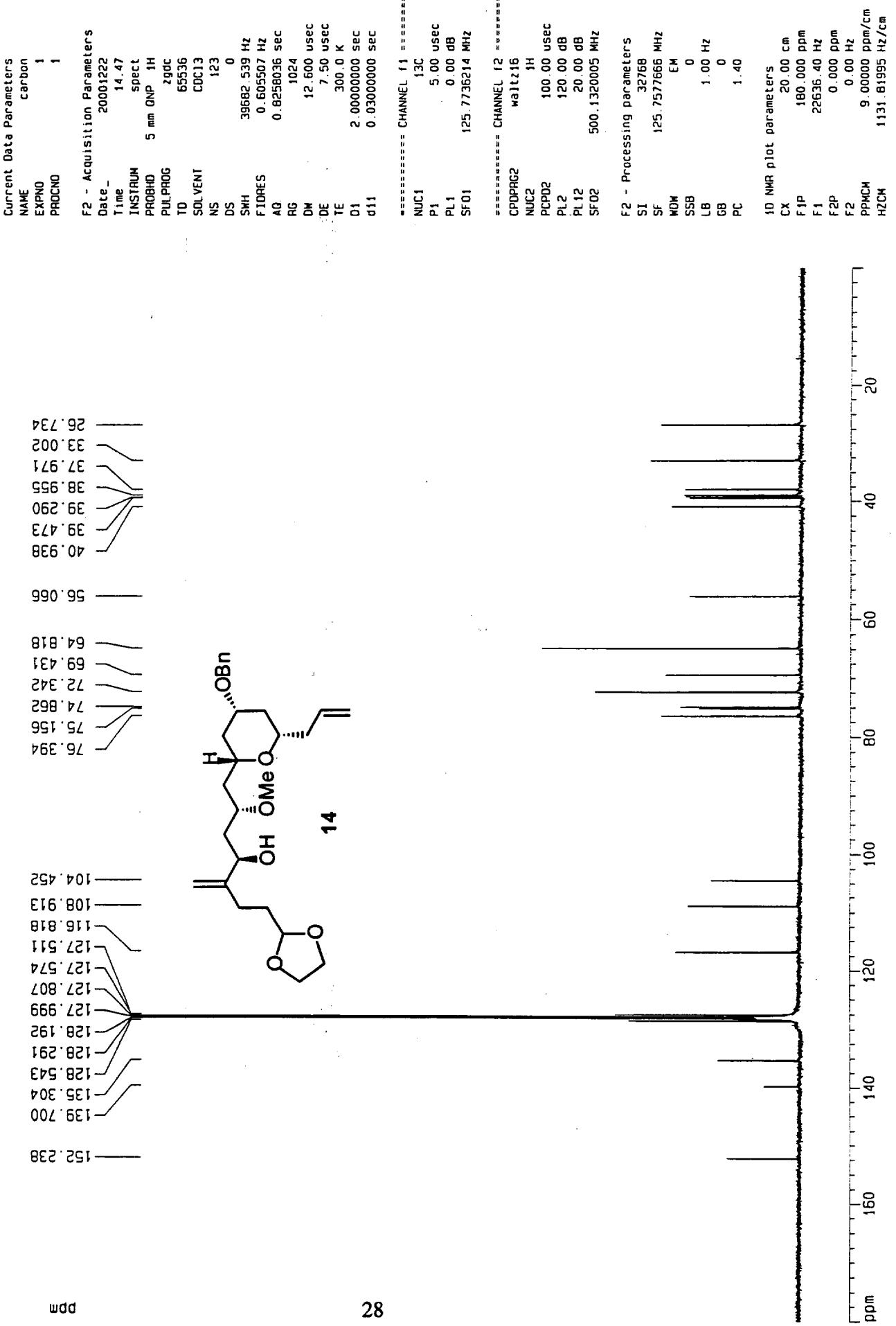

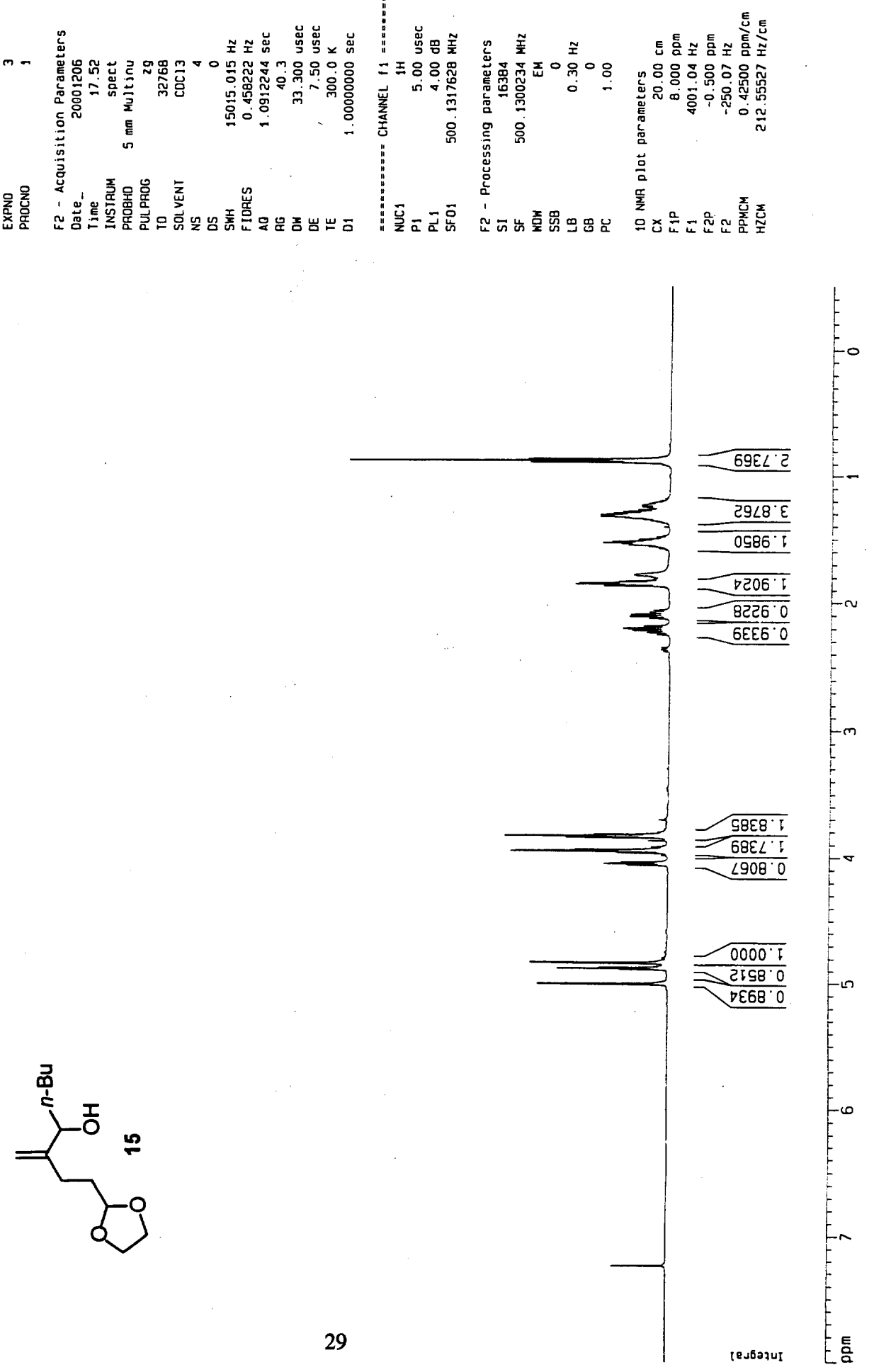

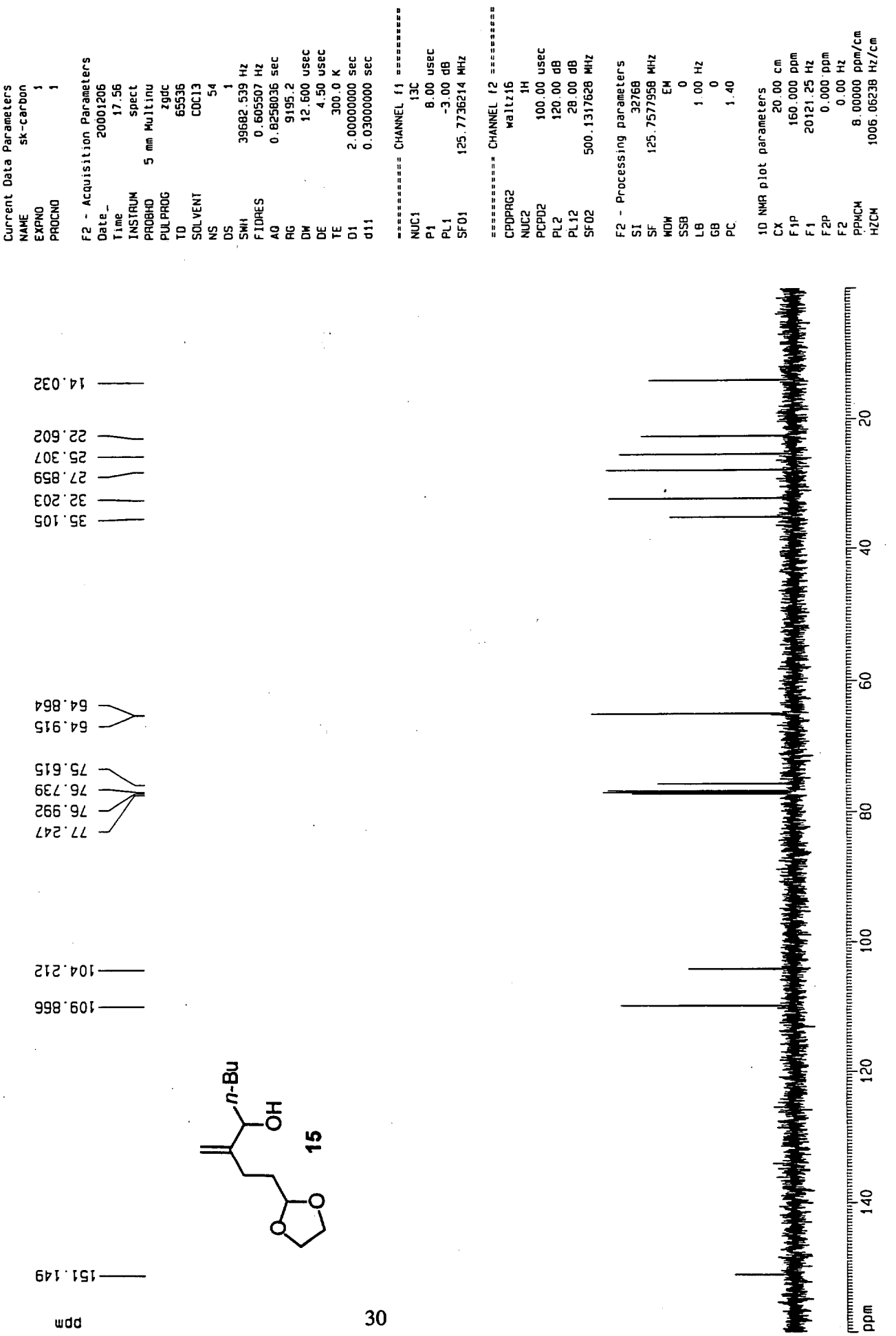

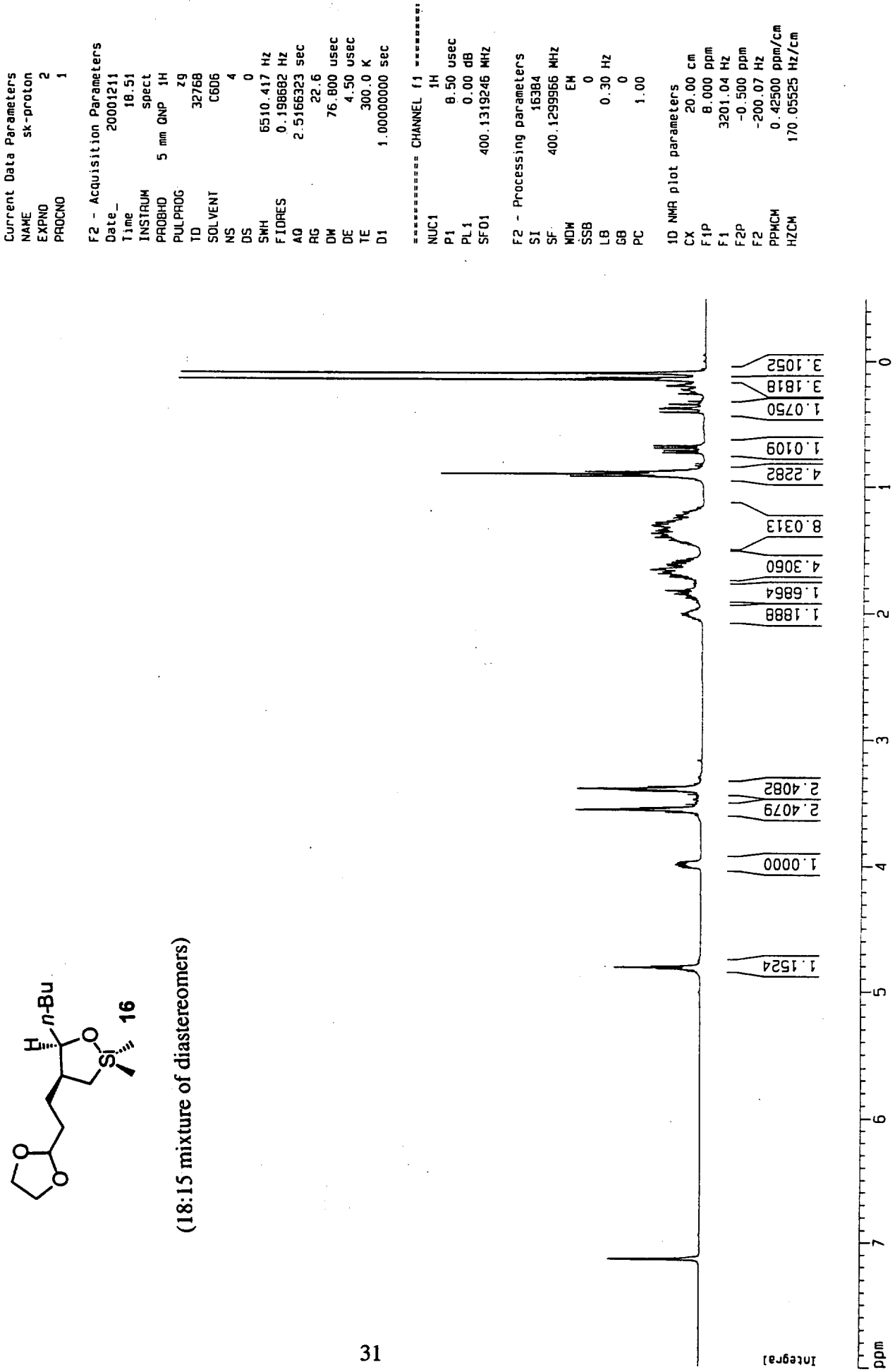

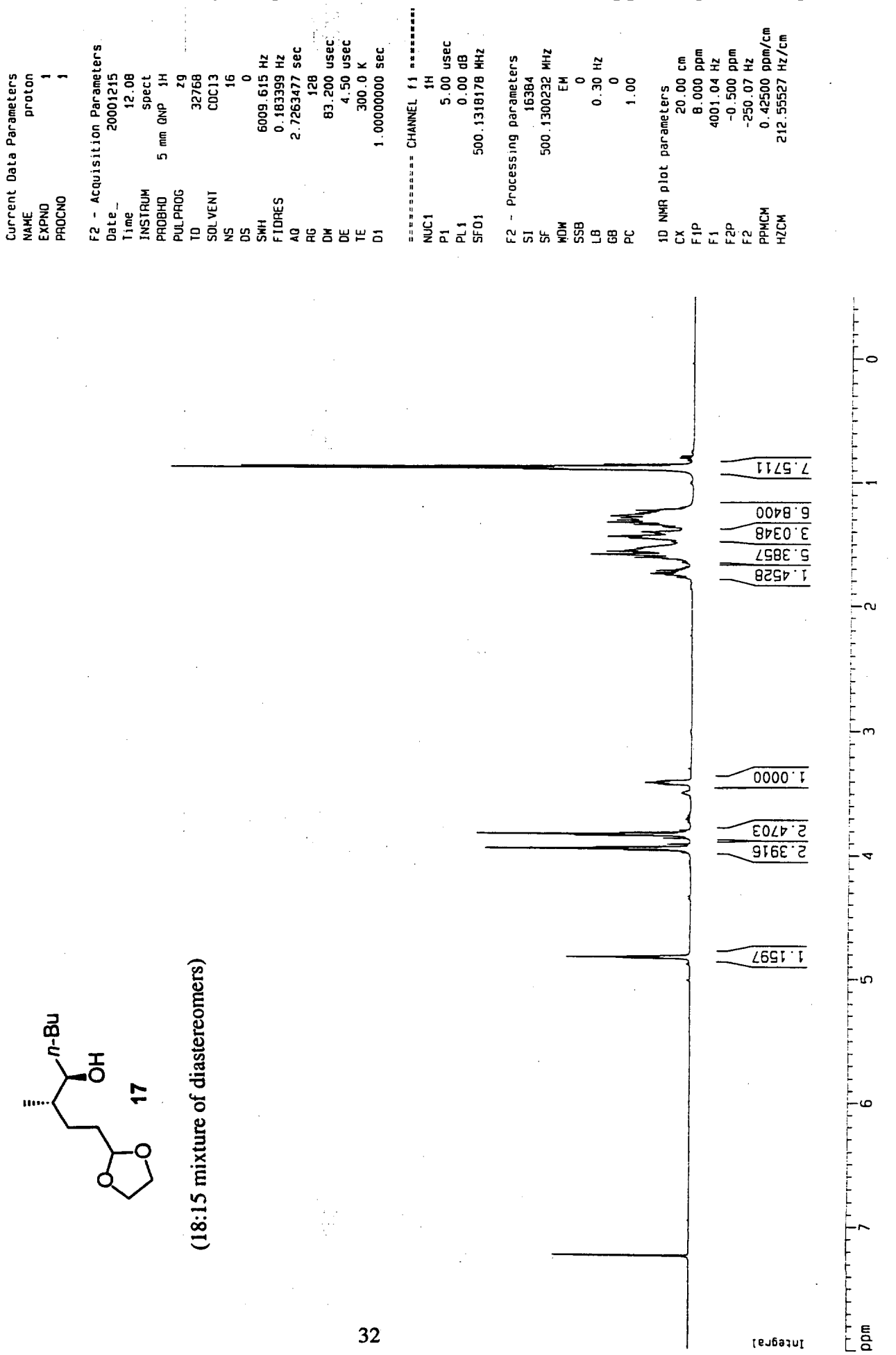

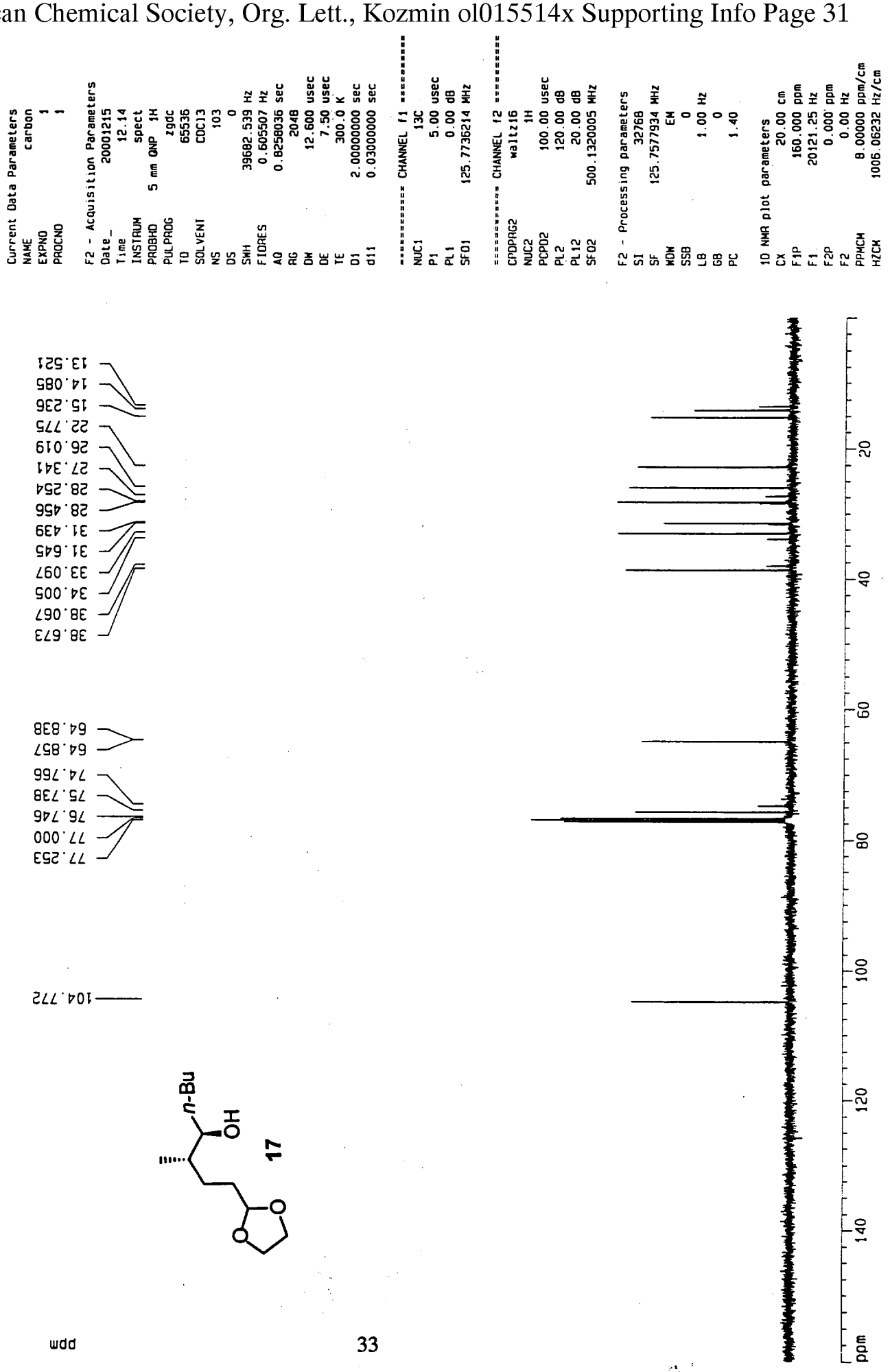

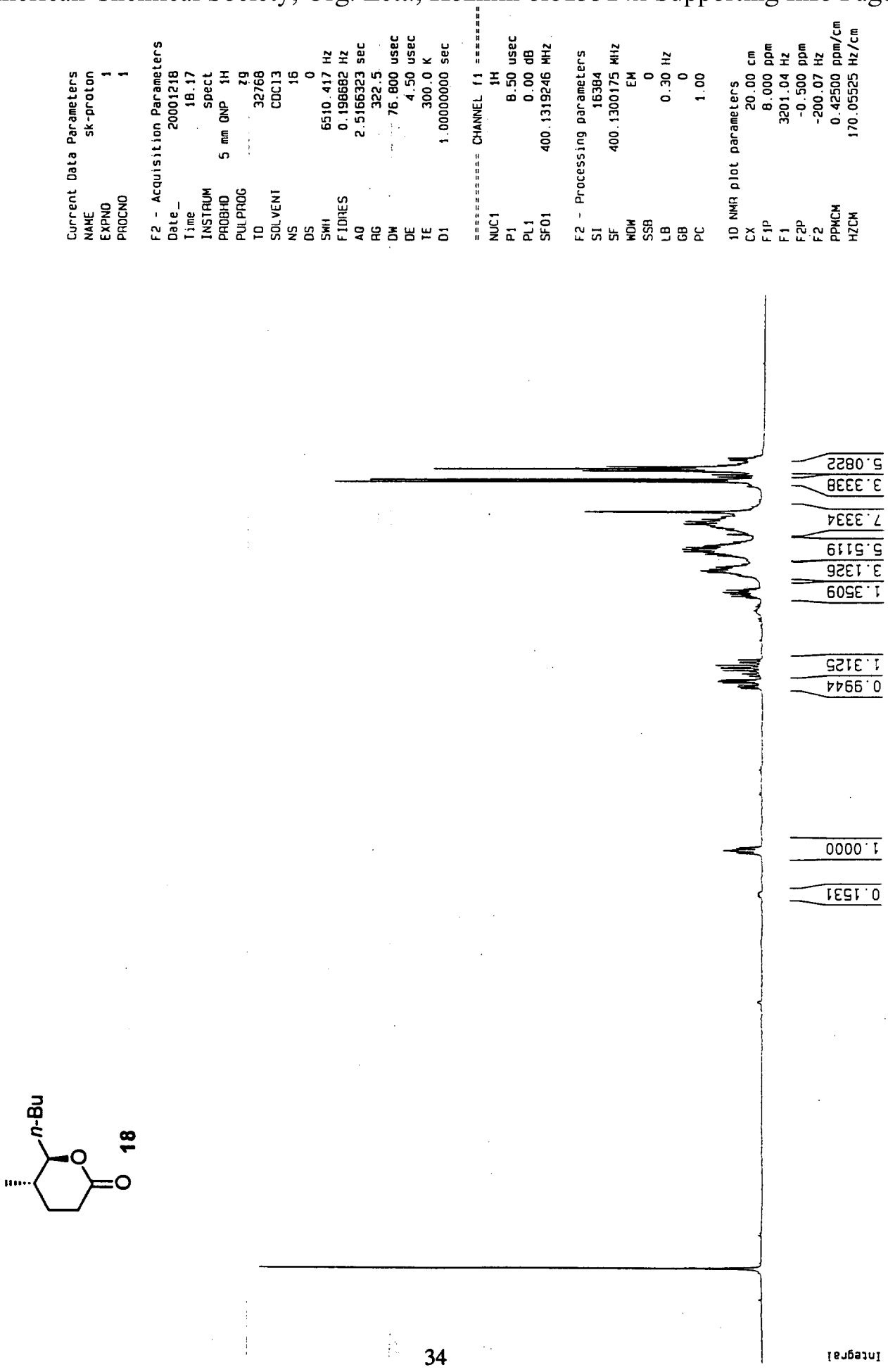

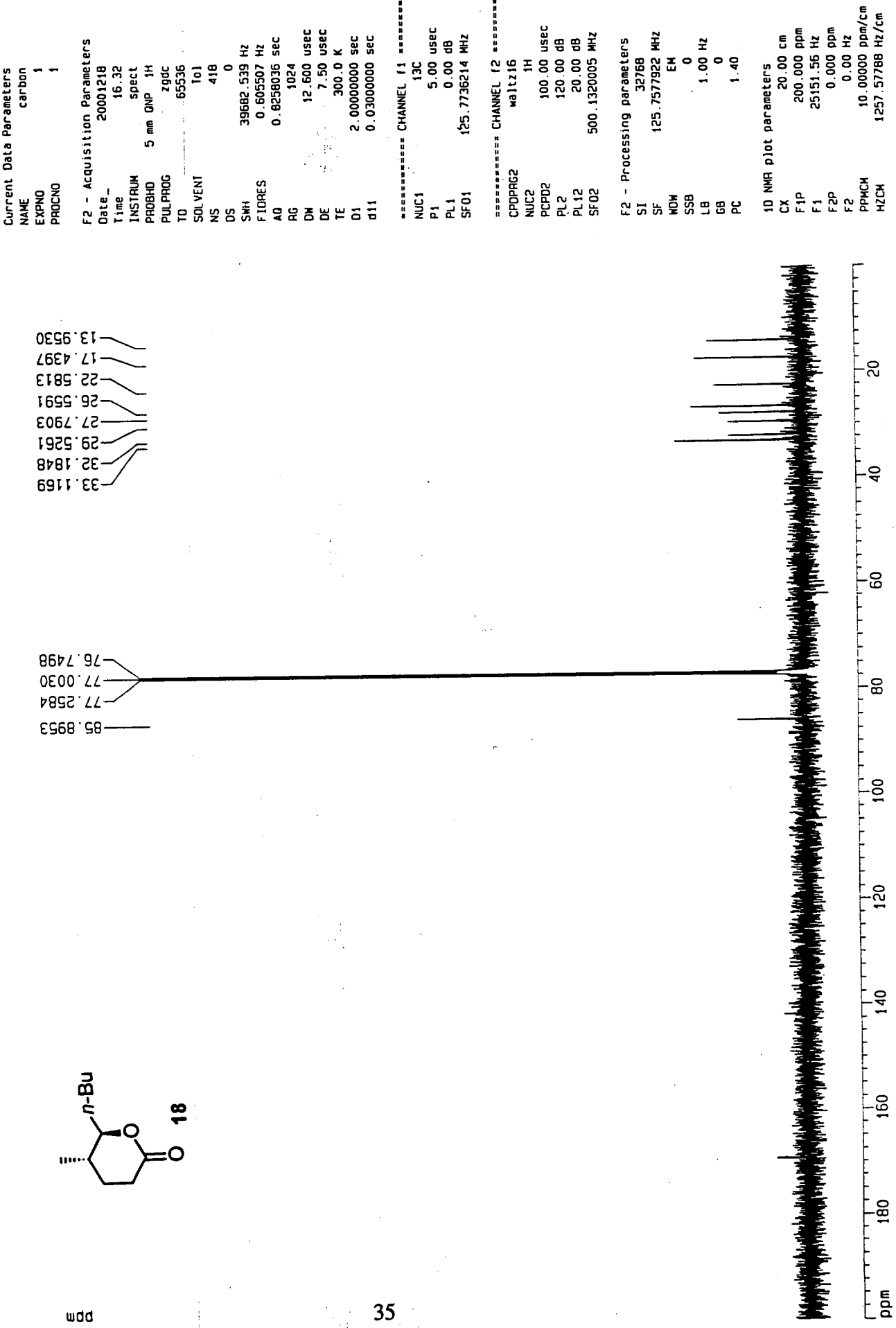

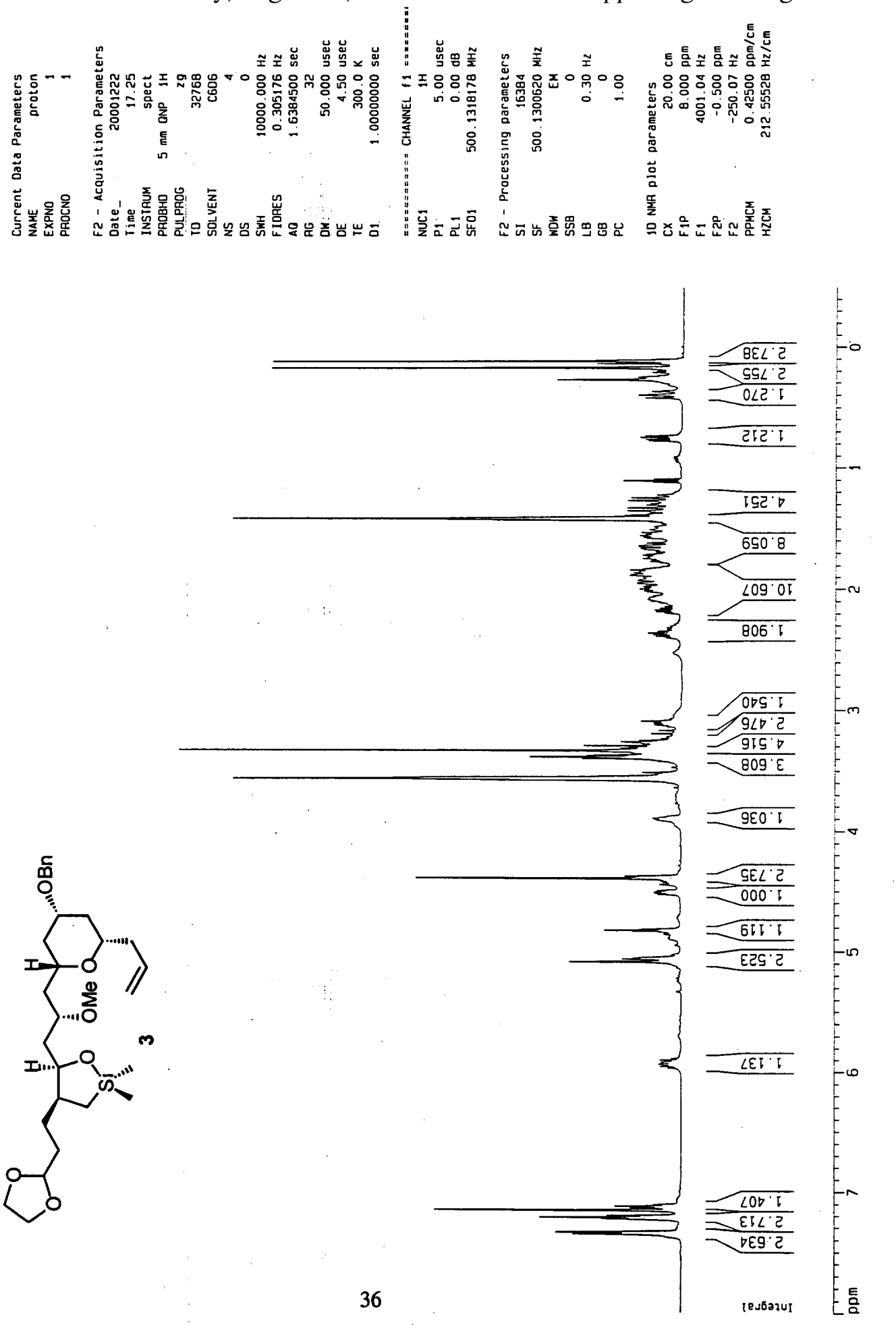

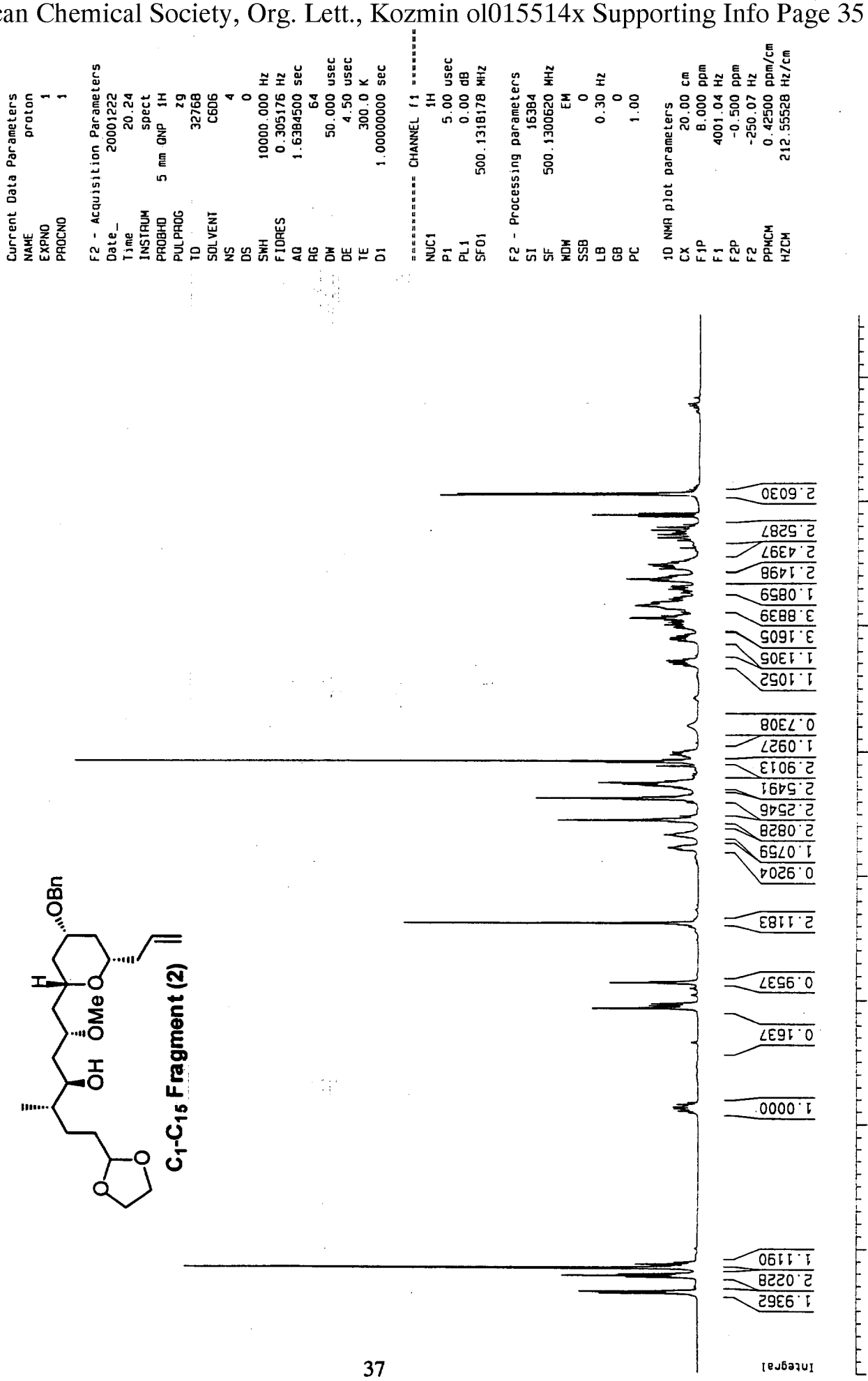

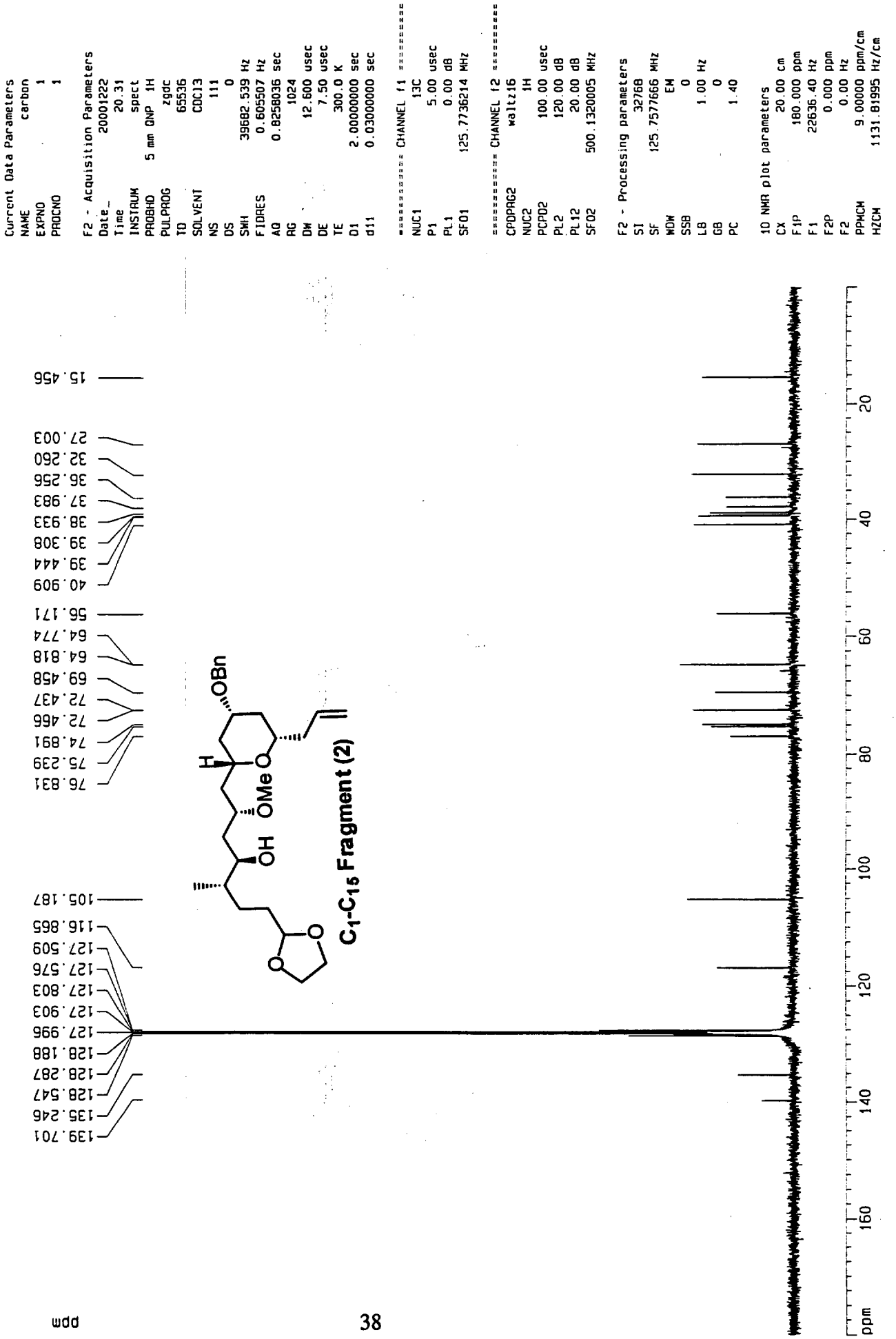
Current Data Parameters
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PROCNO 1
F2 - Acquisition Parameters
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Time 20.31
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PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 111
DS 0
SWH 39682.539 Hz
FIDRES 0.605507 Hz
AQ 0.8258036 sec
RG 1024
DW 12.600 usec
DE 7.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 0.00 dB
SFO1 125.7736214 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 20.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577666 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 22636.40 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.00000 ppm/cm
HZCM 1131.81995 Hz/cm
15.456
27.003
32.260
36.256
37.983
38.933
39.308
39.444
40.909
56.171
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64.818
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72.437
72.466
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75.239
76.831
105.187
116.865
127.509
127.576
127.803
127.903
127.996
128.188
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128.547
135.246
139.701
OBn
H
O
OMe
OH
O
O
C1-C15 Fragment (2)
ppm
20
40
60
80
100
120
140
160
ppm